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National Educators' Workshop: Update 89

Standard Experiments in Engineering Materials Science and Technology

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PREFACE

The need to strengthen materials education is a concern of many in government, industry, and education. In keeping with the theme of the 1989 National Academy of Sciences report, National Research Council's *Materials Science and Engineering for the 1990s: Maintaining Competitiveness in the Age of Materials*, the National Educators' Workshop: Update (NEW: Update) series of workshops has provided over two hundred and sixty materials educators with the latest developments in materials science and engineering while offering them strategies for improved teaching.

These NEW: Update participants saw nearly fifty experiments and demonstrations presented live or on videotape and were provided supplemental notes for replicating the experiments. Peer review and publication of the experiments and demonstrations have provided the materials education community with current, valuable aids for teaching.

NEW: Update 89 served as a forum for presentation of the experiments and information in this publication. The material is a result of years of research aimed at better methods of teaching materials science, engineering and technology. The experiments were developed by faculty, scientists, and engineers throughout the United States. There is a blend of experiments on new materials and traditional materials. Uses of computers in materials science and engineering (MSE), experimental design, and an approach to systematic materials selection were among the demonstrations presented.

More than eighty participants from the United States and Canada who attended NEW: Update 89 observed the experiments and provided critiques for the authors to make modifications prior to this publication. Follow-up activities provided additional resources such as sample materials and videotapes for participants.

NEW: Update 89, and the 86, 87, and 88 workshops are, to our knowledge, the only national workshops or gatherings for materials educators that have a focus on the full range of issues on strategies for better teaching about the full complement of materials. Recognizing the problem of motivating young people to pursue careers in MSE, we have included exemplary pre-university activities such as Adventure in Science, ASM International Education Foundation's Career Outreach Program, and several programs run through high schools.

This year we learned about the Materials Science Technology Project at Richland High School (Richland, Washington) that has received support from Battelle PNL (Pacific Northwest Laboratory). An experiment was presented from the National Science Foundation and AT&T supported program at Science School in Newark, New Jersey. One high school science fair experiment, presented by its student author, had value for MSE education at the college level.

NEW: Update 89, as in past years, involved faculty from community colleges, smaller colleges, and major universities. Participants included those who were the only materials educators on their campus, while others were from well established MSE programs. The Materials Education Council of the United States was represented again and will publish selected experiments in the *Journal of Materials Education*. As with previous NEW: Updates the latest developments in materials research and development were presented by scientists from industry. Tours of NASA Langley labs provided a firsthand view of the latest research in materials science.

NEW: Update 89, with its diversity of faculty, industry, and government MSE participants, served as a forum for both formal and informal issues facing MSE education that ranged from the problems with competition for laboratory time and space to outmoded equipment and the threat by administrators to close laboratories. There were discussions on means to include politicians and policy makers in our collective efforts to help the United States become more competitive through strengthened materials education. We looked at existing and emerging degree programs for technicians for MSE and a model curriculum for such programs.

NEW: Update 89 resulted from considerable cooperative efforts by individuals in government, education, and industry. The workshop goal is to maintain the network of participants and to continue to collect these ideas and resources to bring together a comprehensive manual of standard experiments in materials science, engineering and technology.

NEW: Update 90 will be held November 13-15, 1990 on the campus of the National Institute of Standard and Technology (NIST) in Gaithersburg, Maryland. As with previous workshops, the theme will be strengthening materials education. Participants will witness demonstrations of experiments, discuss issues of MSE with people from education, industry, government, and technical societies, hear about new MSE developments, and tour state-of-the-art NIST laboratories.

We hope that the experiments presented in this publication assist you in teaching about materials science, engineering and technology. We would like to have your comments on their value and means of improving them. Please send comments to James A. Jacobs, School of Technology, Norfolk State University, Norfolk, Virginia 23504.

We express our appreciation to all those who helped to keep this series of workshops viable.

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CONTENTS

PREFACE	iii
PARTICIPANTS	ix
INDUSTRIAL PLASTICS WASTE: IDENTIFICATION AND SEGREGATION	1
Edward L. Widener—Purdue University	
DIELECTRIC DETERMINATION OF THE GLASS TRANSITION TEMPERATURE (T_g)	7
Heidi R. Ries—Norfolk State University	
DYNAMIC MECHANICAL ANALYSIS OF POLYMERIC MATERIALS	13
Kristen T. Kern and Wynford L. Harries—Old Dominion University and Sheila Ann T. Long—NASA Langley Research Center	
ANODE MATERIALS FOR ELECTROCHEMICAL WASTE DESTRUCTION	19
Peter M. Molton—Battelle, Pacific Northwest Laboratory and Clayton Clarke—Florida A & M University	
A SIMPLE DEMONSTRATION OF CORROSION CELLS	29
Philip J. Guichelaar and Molly W. Williams Western Michigan University	
THERMAL CONDUCTIVITY OF METALS	37
Sayyed M. Kazem—Purdue University	
THE ASSESSMENT OF METAL FIBER REINFORCED POLYMERIC COMPOSITES	45
Wenchiang R. Chung—San Jose State University Presented by Seth P. Eates—San Jose State University	
EXPERIMENTAL DETERMINATION OF MATERIAL DAMPING USING VIBRATION ANALYZER	53
Mostafiz R. Chowdhury and Farida Chowdhury East Carolina University	
RECYCLING WASTE-PAPER	65
Edward L. Widener—Purdue University	

PIEZOELECTRIC AND PYROELECTRIC EFFECTS OF A CRYSTALLINE POLYMER	71
Nikhil K. Kundu—Purdue University and Malay Kundu—John Adams High School	
USING TEMPLATE/HOTWIRE CUTTING TO DEMONSTRATE MOLDLESS COMPOSITE FABRICATION	77
J. Mario Coleman—West Texas State University	
RUBBERLIKE ELASTICITY EXPERIMENT	89
Richard Greet and Robert Cobaugh—University of Southern Colorado	
THE EFFECT OF THERMAL DAMAGE ON THE MECHANICAL PROPERTIES OF POLYMER REGRINDS	93
Nikhil K. Kundu—Purdue University	
DEMO OF THREE WAYS TO USE A COMPUTER TO ASSIST IN LAB	101
J. P. Neville—Wentworth Institute of Technology	
THE MAGNETIZATION PROCESS—HYSTERESIS	105
Richard Balsamel—Science High School, Newark, NJ	
TENSILE AND SHEAR STRENGTH OF ADHESIVES	115
Kenneth A. Stibolt—Anne Arundel Community College	
EXPERIMENTS AND OTHER METHODS FOR DEVELOPING EXPERTISE WITH DESIGN OF EXPERIMENTS IN A CLASSROOM SETTING	119
John W. Patterson—Iowa State University	
MANUAL AND COMPUTER-AIDED MATERIALS SELECTION FOR INDUSTRIAL PRODUCTION: AN EXERCISE IN DECISION MAKING	133
Seth P. Bates—San Jose State University	
SCANNED-PROBE MICROSCOPES	141
H. Kumar Wickramasinghe—IBM Research Division Presented by F. Alan McDonald—IBM Research Division	
PREPARING TECHNICIANS FOR ENGINEERING MATERIALS TECHNOLOGY	143
James A. Jacobs—Norfolk State University and Carlton H. Metzloff—Erie Community College—North	

FUTURE AUTOMOTIVE MATERIALS—EVOLUTION OR REVOLUTION	151
P. Beardmore—Ford Motor Company	
AUSTEMPERING	171
James P. Nagy—Erie Community College	
HANDS-ON THERMAL CONDUCTIVITY AND WORK-HARDENING AND ANNEALING IN METALS	177
L. Roy Bunnell—Battelle, Pacific Northwest Laboratories	
Presented by Stephen W. Piippo—Richland High School	
CERAMIC FIBERS	181
Bruce Link—Corning Glass	

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INDUSTRIAL PLASTICS WASTE: IDENTIFICATION AND SEGREGATION

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ABSTRACT

Throwaway plastics products, mainly packaging, are inundating our landfills and incinerators. Most are "ethenic" thermoplastics, which can be recycled as new products or fossil-fuels. Lab experiments are described, involving destructive and non-destructive tests for identifying and using plastics. The burn-test, with simple apparatus and familiar samples, is recommended as quick, cheap and effective.

INTRODUCTION

Our throwaway society is generating a hundred-billion dollar waste-disposal industry as costs approach a hundred dollars per ton. Programs to segregate and salvage metal-paper-glass are commonplace, albeit under-utilized. Programs to recycle plastics are rare, although the bulky films-fibers-foams are littering landscapes and choking landfills. In the U.S.A., seventy-five percent of all trash tonnage and ninety-nine percent of all plastics waste now go to landfills. This includes five million tons (907 kg/T) of dirty diapers/year. By 1990, about one sixth of 1960 landfills will be open, yet trash volume will have doubled. Environmental activists have stymied most plans for new landfills and incinerators. Simultaneously protecting our ecology and preserving our economy is a challenge. Technology's task is to simplify the admittedly complex subject of polymer chemistry, to include plastics in the curriculum, to establish realistic priorities, to publicize an optimal solution to interactive problems, and to promote public consensus rather than conflict.

PROBLEMS

Plastics are polymers (giant molecules) from fossil-fuels (coal, oil, gas) or from bio-mass (animal, vegetable). It seems foolish to mix irreplaceable and priceless fuels with garbage, or to embalm degradable compost in sealed hi-tech landfills. Yet government regulations and class-action lawsuits await the unwary. At present, the salvaging of plastics mainly involves recycled scrap (in-house) and remolded products (low-quality). Waste-to-energy plants, that vast market for mixed-plastics trash, are publicly seen as "incinerators". Polluters of land (toxic ash), water (leachate), and atmosphere (acid-rain, ozone-depletion, smog) are banned. Yet, everyone pollutes the environment.

Inherently, plastics are glassy and amorphous, thus low-density (0.9-2.0 g/cc). Being non-conducting and non-magnetic, they hold static charges. Thermal expansion and moisture-absorption bring curling and clinging problems, which hinder compacting and handling. Although materials are generally formulated as w/o (percent by

weight), the waste plastics are better described in v/o (percent by volume). Some 30-50v/o of municipal trash is plastics, mainly from consumer packaging.

Most polymers are carbon compounds (organic) or silicon compounds (inorganic) with only six other elements (chlorine, fluorine, hydrogen, nitrogen, oxygen, sulfur). Possible polymers are myriad, considering isomers-copolymers-terpolymers-alloys and elastomers; but the usual plastics waste-stream only consists of a few "vinyls" (ethenics) as solid-liquid-gas in many forms (block, sheet, fiber, cloth, felt, film, or foam). Natural colors are clear-cloudy-white, subsequently contaminated with dyes, or pigments, and readily degraded to yellow-orange-brown-black by radiations (ultra-violet, infra-red, gamma). Mechanical properties (strength, stiffness, hardness) can vary widely (with structure and additives). Melting point is a misnomer, when plastics are "super-cooled" liquids; better terms are "softening or molding" ranges, with upper and lower "glass-transition" temperatures. True crystallinity is best attained in "thermosets" (cross-linked networks) with strong co-valent bonding; but partial-crystallizing occurs in "thermoplastics" (drawn or molded linear-chains) with weak surface-bonding. Mers can make plain "aliphatic" chains or be linked as "aromatic" rings. The same name may apply to many polymers; several names may apply to the same polymer. Tests can help to dispel such confusion.

EXPERIMENTS

Several simple methods for identifying plastics types are recommended:

1. Plastikit I (25-thermoplastics) and Plastikit II (25-thermosets) are boxed sheets (10cm x 10cm, 5 commercial thicknesses) and were designed to SPE specifications for non-destructive tests (stiffness, clarity, moldability, flatness, expansion, density, moisture pickup, color). Also, spot checks of solvents, adhesives, stains and heat are feasible. Optimal stiffener-fins (1-1/2 t high, 2/3 t wide, 4cm long) are molded on the end of each thickness (t). Shrinkage marks (1-inch flow-direction, 2-inch cross-direction) are included for thin grades (30,50,70 and 90-mils; 1,000 mils/inch, 2.54 cm/inch).
2. Resinkit (43-thermoplastics) in notebook format was designed to SPE specifications for destructive tests (brittleness, toughness, burning). Samples look like combs (10cm long) with 14-teeth (each 2mm x 2mm x 2cm) formed at each end (28 possible burn-tests). A descriptive card for each plastic also describes its properties and processibility. These are used as "control" samples for comparison with unknown plastics specimens. Most unknowns will be glassy thermoplastic ethenics.
3. Unknown plastics waste may then be subjected to destructive and non-destructive tests. Common sources are
Polystyrene-Trays, bowls, dishes, cups, covers, spoons (from restaurants and grocery stores); insulation (building panels).

Polyethylene-Milk or cider jugs, pipe caps, pop-bottle bases, 6-pack yokes, spun-bonded fabrics and envelopes.

Polyester-Pop bottles (thermoplastic PET); corrugated siding or roofing (thermoset FRP panels).

Polyvinyl Chloride-Synthetic leather (gloves, bags, shoes, chairs); hard pipes or fittings (white, grey, green).

Polyurethane-Yellow foam (packaging, insulation, rubber toys).

Polymethyl Methacrylate-Clothing (fibers) and lenses (auto).

Phenol Formaldehyde-Electric plugs, distributor caps, hot pan handles.

Melamine Formaldehyde-Plastic dishes (T/S, thick walls).

Elastomers-Tires, tubes, belts, rubber bands, balloons, and balls.

Silicones-Caulking strips and tubes.

Fluoro-carbons-Pipe thread tapes, pan coatings, anti-friction parts (white, grey).

Typical mechanical tests for stiffness, clarity, color, flatness, hardness, toughness and fold-endurance are first. Follow these with softening tests (with solvents, boiling water, soldering iron, hot-plate, or oven). Finally, conduct a "burn-test", observing flame, stickiness, smoke, smell and residue.

4. Burn Testing

Because plastics are essentially "low-temperature" materials, exposure to flame is probably the quickest and cheapest way to identify common samples of waste. Such tests are usually destructive, hazardous, and dramatic. Get small samples (say 1cm x 3cm x 2mm); fire-proof surface (ash-tray, pie-tin); controlled flame (match, candle, lighter, burner); sample holder (paper clip, spring clamp, tweezers, tongs); fire extinguisher (water bucket); and ample ventilation (hood).

Hold each sample at a 45-degree angle (film or foam may need compacting) and light the bottom edge. Remove flame and judge the combustibility (increased, stable, decreased, self-extinguished). Touch sample to pan and observe stickiness, bubbling, dripping, or charring. Watch the flame-base to see colors (tip usually yellow; base may be blue, purple, green, orange). Gently blow out flame (no splatter) and observe smoke (much or little; grey-black-white; soot). Gingerly waft the fumes toward your nose (do not inhale noxious fumes, smoke or flame) and describe the odor. Taste and smell are tricky; use imagination; breathe fresh air and drink water, between samples; emotional and physical states may influence perceptions. Do not touch hot samples. Repeat tests if needed (no large samples).

Finally, refer to a published "burn-chart" (see Dietrich Braun, "Identification of Plastics", ref. 10; contact technical-service representatives at Dow, DuPont, Hercules, or Shell Corporations).

Typical results are

1. Organic plastics support combustion. Thermosets are safer than thermoplastics. Sheets burn slower than films, which are slower than foams.
2. Styrene burns fiercely, has orange flame and sooty smoke, smells like marigolds, softens-drips-spurts.
3. Urethane burns like styrene, but has light yellow flame, less smoke, apple smell.
4. Ethylene feels waxy, burns blue like methane gas, smells and drips like a candle. Propylene is similar, but stiffer; better fatigue-life; whiter smoke.
5. Vinyl chloride is shiny, hard to light, green flame and white smoke, chlorine smell.
6. Fluorocarbon is hard to light, green flame, little odor or smoke.
7. Nylon is medium-burning, blue flame, smells like singed hair or burned wool.

ABBREVIATIONS (Tradename typical)

- | | | | |
|-----|------|---|--|
| 1. | CPVC | - | Chlorinated PVC, like PVDC |
| 2. | CR | - | Poly Chloroprene Rubber (<u>Neoprene</u>) |
| 3. | FRP | - | Fiberglas Reinforced Polyester (T/S) |
| 4. | IIR | - | Poly Isoprene - Isobutylene Rubber (Butyl) |
| 5. | IR | - | Poly Isoprene Rubber (<u>Latex</u>) |
| 6. | MF | - | Melamine Formaldehyde (<u>Mel-mac</u>) |
| 7. | NBR | - | Poly Butadiene - Acrylonitrile Rubber (Buna N) |
| 8. | PA | - | Polyamide (<u>Nylon</u>); Aramid (<u>Kevlar</u> , <u>Nomex</u>) |
| 9. | PAI | - | Polyamide/imide (<u>Torlon</u>) |
| 10. | PC | - | Polycarbonate (<u>Lexan</u>) |
| 11. | PE | - | Poly Ethylene (Ethene, Olefin, <u>Tyvek</u>) |
| 12. | PET | - | Poly Ethylene Terephthalate (T/P Polyester, <u>Dacron</u> , <u>Mylar</u>) |
| 13. | PF | - | Phenol Formaldehyde (<u>Bakelite</u>) |
| 14. | PI | - | Polyimide (<u>Kapton</u>) |
| 15. | PMMA | - | Poly Methyl Methacrylate (<u>Lucite</u> , <u>Plexiglas</u> , <u>Orlon</u>) |
| 16. | PP | - | Poly Propylene (Propene) |
| 17. | PS | - | Poly Styrene |
| 18. | PTFE | - | Poly Tetra Fluoro Ethylene (<u>Teflon</u>) |
| 19. | PUR | - | Poly Urethane Rubber |
| 20. | PVC | - | Poly Vinyl Chloride |
| 21. | PVDC | - | Poly Vinylidene Chloride (<u>Saran</u>) |
| 22. | SBR | - | Styrene Butadiene Rubber (<u>Buna S</u>) |
| 23. | SI | - | Silicone (silane, siloxane, <u>RTV</u>) |
| 24. | SPE | - | Society of Plastics Engineers |
| 25. | SPI | - | Society of Plastics Industries |
| 26. | T/P | - | Thermoplastics |
| 27. | T/S | - | Thermosets |
| 28. | UF | - | Urea Formaldehyde |

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BIOGRAPHY

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DIELECTRIC DETERMINATION OF THE GLASS TRANSITION TEMPERATURE (T_g)

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OBJECTIVE

To determine the glass transition temperature of a polymer using a dielectric dissipation technique.

EQUIPMENT

Polymer specimen, aluminum foil electrodes, optically flat pyrex discs, brass weight, thermocouple, thermal joint compound, furnace, minicomputer, plotter, LCR meter (inductance, capacitance, resistance meter)

BACKGROUND

The "glass transition" occurs in materials which can be cooled from a liquid phase to a solid phase without crystallizing. As a viscous liquid is cooled, its viscosity increases with decreasing temperature. The point at which the viscosity becomes so large that the molecules are rigidly fixed in place, although no crystallization has occurred, is considered to be the glass transition temperature (T_g). Thus, below T_g the molecules are "frozen" in place, whereas above T_g the molecules are relatively free to move. A large variety of materials exhibiting a glass transition exist, including organic polymers, inorganic oxides, molecular liquids, fused salts and some metallic systems [1].

The glass transition between the solid phase and the liquid phase results in changes in a variety of properties of the material. In this experiment, the change in the dielectric dissipation factor of the material will be measured in order to determine T_g .

The dielectric dissipation factor is a measure of how much energy is dissipated when an alternating electric field is applied across a capacitor. Although the dissipation factor has several components, the contribution from the dipolar molecules in the material is the piece which varies significantly with temperature.

*This experiment was developed at NASA Langley Research Center. The author worked with this equipment under NASA Cooperative Agreement NCC1-90.

In a dielectric material below T_g , the molecules are "frozen" in place, cannot respond to the applied field, and therefore do not contribute to the dissipation of energy. Consequently, the dissipation factor is relatively constant for all temperatures below T_g .

As the temperature is increased above T_g , however, the viscosity decreases, so the molecules may gradually begin to move in response to the applied field. Since the molecules are not completely free to move at this point, they will lag somewhat behind the applied field, resulting in the dissipation of energy. Therefore, the dissipation factor will increase significantly at T_g .

At temperatures well above T_g , the viscosity will decrease so much that the molecules will be free enough to move in phase with the electric field. When the molecules are in step with the electric field, energy will not be dissipated by this means, so the dissipation factor will decrease.

SUMMARY

In summary, a peak in the dissipation factor versus temperature curve is expected near the glass transition temperature T_g , as shown in Figure 1. It should be noted that the glass transition is gradual rather than abrupt, so that the glass transition temperature T_g is not clearly identifiable. In this case, the glass transition temperature is defined to be the temperature at the intersection point of the tangent lines to the dissipation factor versus temperature curve above and below the transition region, as illustrated in Figure 1.

In this experiment, the dielectric dissipation factor technique will be applied to polymers.

PROCEDURE

- 1) Stack the apparatus as shown in Figure 2.

Typically, the specimen will be 5.5 cm in diameter and the other components are 5.0 cm in diameter.

The electrodes may be cut from aluminum foil, with "tails" attached for leads to the LCR meter. Be sure that they are not wrinkled.

The disks and brass weight are present to insure uniform pressure at the electrode-specimen interface.

Thermal joint compound is used at the tip of the thermocouple to provide contact with the quartz disk.

- 2) Place the assembly in the furnace. Connect the leads to the LCR meter, and begin data collection. (The total temperature range and temperature intervals for data collection will depend upon the sample you are using.)

CAUTION! DO NOT BURN YOURSELF!

- 3) Analyze the data. Determine the best line fits to the curve above and below the transition region, as shown in Figure 1. The glass transition temperature is the temperature at the intersection of these two lines.

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INSTRUCTOR'S NOTES

When this experiment was performed at NASA Langley Research Center, the data acquisition system was controlled by an HP-9830A computer. The data acquisition program reads data from the thermocouple to determine the specimen temperature, and then reads the dissipation factor measurements from an HP-4275A multifrequency LCR meter at temperature intervals and frequencies specified by the operator. The furnace was adjusted to provide a heating rate of 6 °C/min. It was found that this heating rate and a frequency of 10 kHz yields T_g measurements which are in agreement with measurements obtained from other techniques.

The data was analyzed using a linear regression program to determine the best line fits. The computer was then used to calculate the temperature at the intersection of the two lines.

If a computer is not available to control the experiment, the student could record the temperature and dissipation factor readings manually and perform the data analysis by hand. Variations of only two or three degrees are typically introduced when the data is analyzed manually.

ADDITIONAL NOTES

As discussed previously, the glass transition temperature occurs when the molecules become free to move in the material. Thus, T_g is dependent upon the length of the polymeric chain, and therefore upon the processing techniques used.

Measurement of the glass transition temperature is therefore useful in comparing the degree of crosslinking induced by various processing techniques. For example, irradiation of the polymer

film may result in crosslinking or chain scissioning, with a corresponding increase or decrease in T_g .

Thus, this T_g experiment may be used in conjunction with the study of a variety of polymer processing techniques.

It should also be noted that some polymers which possess pendant groups may exhibit more than one transition. This occurs because the pendant group may become free to move at a temperature lower than the temperature at which the entire chain is freed.

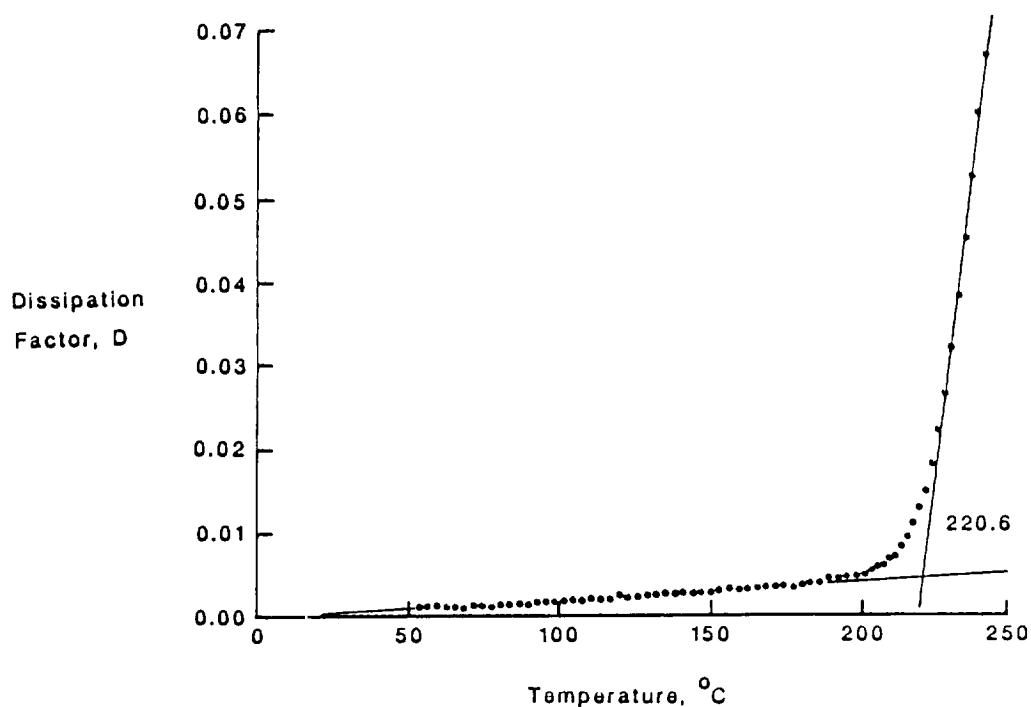


Figure 1. Determination of the glass transition temperature of a polymer from AC electrical dissipation data.

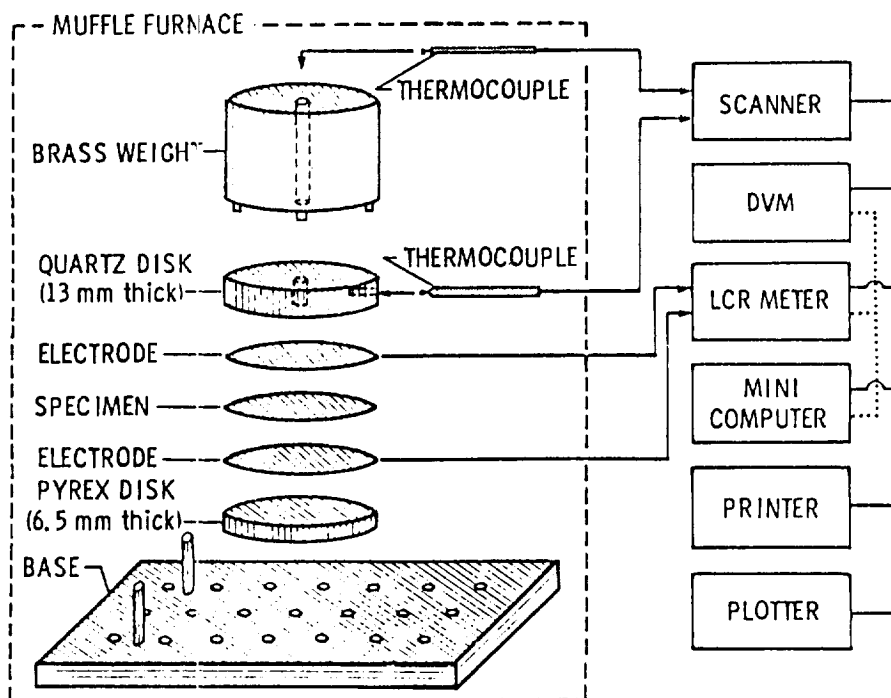


Diagram by: F. R. Long, Jr.
NASA-LRC

Figure 2. Experimental arrangement for glass transition temperature measurements.

DYNAMIC MECHANICAL ANALYSIS OF POLYMERIC MATERIALS

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ABSTRACT

Polymeric materials exhibit mechanical behavior which is dependent on temperature. Dynamic mechanical analysis measures the mechanical damping and resonant frequency of a material over a temperature range. Values of the dynamic loss modulus, storage modulus, and loss tangent can be calculated from these data. The glass transition temperature and onset temperature are obtained from curves of the dynamic moduli versus temperature.

INTRODUCTION

Polymeric materials do not deform easily at low temperatures. This is referred to as the glassy state. At high temperatures, the same material will be rubbery and will deform easily. The temperature at which this change in behavior occurs is called the glass transition temperature, T_g . The transition usually occurs over a temperature range, called the glass transition region. Materials are structurally sound at temperatures below the onset to the glass transition region, hence the importance of determining the glass transition temperature and the onset temperature.

The response, or strain, of a polymeric material to a sinusoidal stress is characterized by a modulus. The modulus, the ratio of stress to strain, is composed of the storage modulus and the loss modulus. The storage modulus is related to the amount of energy stored in the material as a deformation and returned to the oscillation, while the loss modulus is related to the amount of energy lost through friction.

When the temperature is low (glassy state), the loss modulus is small and the storage modulus is large. As the temperature increases, the intermolecular friction changes and the loss modulus increases (transition region) to a maximum (T_g). At still higher temperatures, the loss modulus decreases (rubbery state), and the storage modulus is also small.

Dynamic mechanical analysis of polymeric materials is analogous to the simplified, linear model shown in Figure 1. The equation of motion for this damped, driven harmonic oscillator is

$$M \frac{d^2x}{dt^2} + B \frac{dx}{dt} + Kx = F_0 \sin \omega t$$

where M is the mass of the oscillating body, B is the coefficient of friction, K is the spring constant, x is the displacement of the mass from its equilibrium position, and F_0 is the magnitude of the applied sinusoidal force. For oscillation at resonant frequency, the position of the mass is 90° out of phase with the applied force, and can be written

$$x = x_0 \cos \omega t$$

where x_0 is the maximum displacement. Substituting this expression with its first and second derivatives into the differential equation of motion and comparing the coefficients of the sine and cosine terms, we obtain the equalities

$$K = M\omega^2$$

and

$$\omega B = -F_0/x_0$$

The first of these equations shows that the spring constant, which represents the ability of the oscillation mechanism to store energy, is proportional to the square of the resonant frequency. This is analogous to the storage modulus of a material. The second equation shows that the damping term, representing the loss of energy from the system, is proportional to the applied force and inversely proportional to the magnitude of the oscillation. This is analogous to the loss modulus of a material.

EXPERIMENTAL

The DuPont¹ 982 Dynamic Mechanical Analyzer (DMA) is used to measure the mechanical damping and resonant frequency of a sample of material as a function of sample temperature. The sample is clamped between the ends of two rigid arms, each of which is free to oscillate around a pivot point (See Figure 2). An electromagnetic driving mechanism is connected to the opposite end of one arm. The driving frequency is varied by the apparatus until the driving moment, sensed from the driving current, is a minimum as a function of frequency, provided the amplitude is fixed. This frequency is the resonant frequency and the apparatus adjusts to the resonant frequency which changes with temperature. The oscillation amplitude is monitored by a linear variable differential transformer, which provides a feedback signal to the driving mechanism to maintain a constant amplitude. This same feedback signal, in millivolts, is recorded as a measure of energy lost during each cycle or the damping due to the specimen. With the use of liquid nitrogen, the system can record the resonant frequency and damping of a material from -150°C to 500°C . See Figure 3.

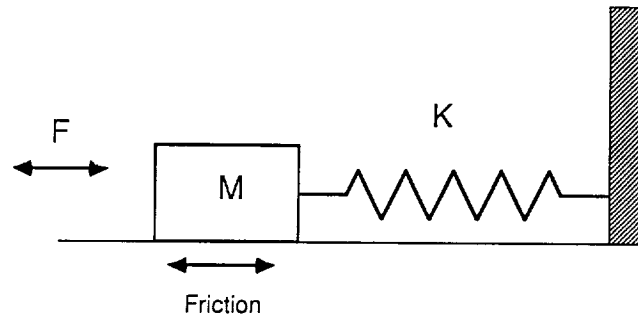
The 982 DMA is accompanied by the 1090 Thermal Analyzer (TA), shown in Figure 4, which is used for data acquisition, data analysis, and temperature control. Data analysis programs provided by DuPont calculate the loss and storage moduli from damping and frequency values stored by the 1090 TA. The frequency and damping signals of the arms with no sample and with a high modulus, low loss material, such as steel, are used to calibrate the instrument.

ANALYSIS

An example of the resulting curves for a sample of T300/934 graphite/epoxy composite is shown in Figure 5. The transition onset temperature is found by drawing tangents to the loss modulus curve before the transition region and during the increasing part inside the transition region, as shown in Figure 5. The intersection of these two tangents is considered to occur at the onset temperature. The glass transition temperature is taken to be the temperature at which the loss modulus reaches a peak.

CONCLUSION

The data shows that the glass transition temperature is 240°C ($\pm 5^{\circ}\text{C}$) for T300/934 graphite/epoxy composite. The onset temperature is 218°C ($\pm 5^{\circ}\text{C}$), below which the material would be structurally sound.



$$M \frac{d^2x}{dt^2} + B \frac{dx}{dt} + Kx = F_0 \sin \omega t$$

For oscillation at resonant frequency,

$$x = x_0 \cos \omega t$$

$$K = M\omega^2$$

$$\omega B = -F_0/x_0$$

Figure 1. The simplified linear model of dynamic mechanical analysis. A mass M attached to a spring of elasticity, K , is driven by an applied sinusoidal force, F . Energy is lost from the oscillation by friction.

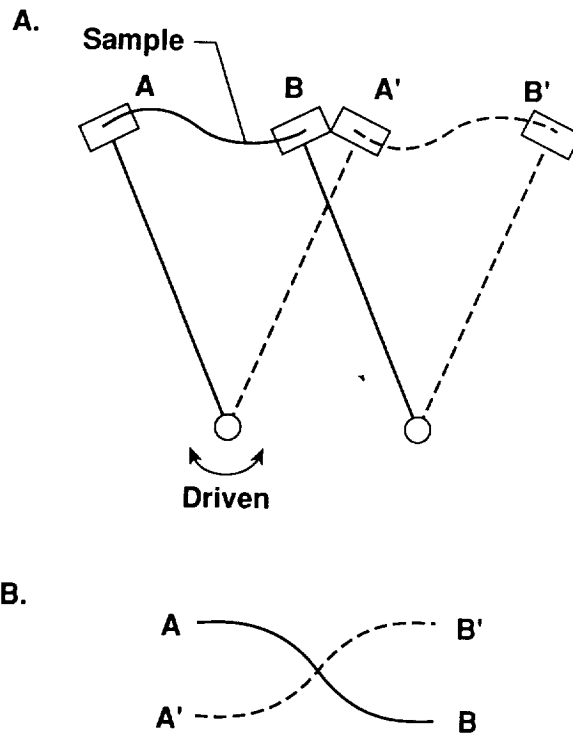


Figure 2. The dynamic mechanical analyzer. A. The oscillation mechanism consists of two arms with a driving mechanism. The sample is clamped at the ends of the arms. B. The sample is flexed in the manner shown (exaggerated here).

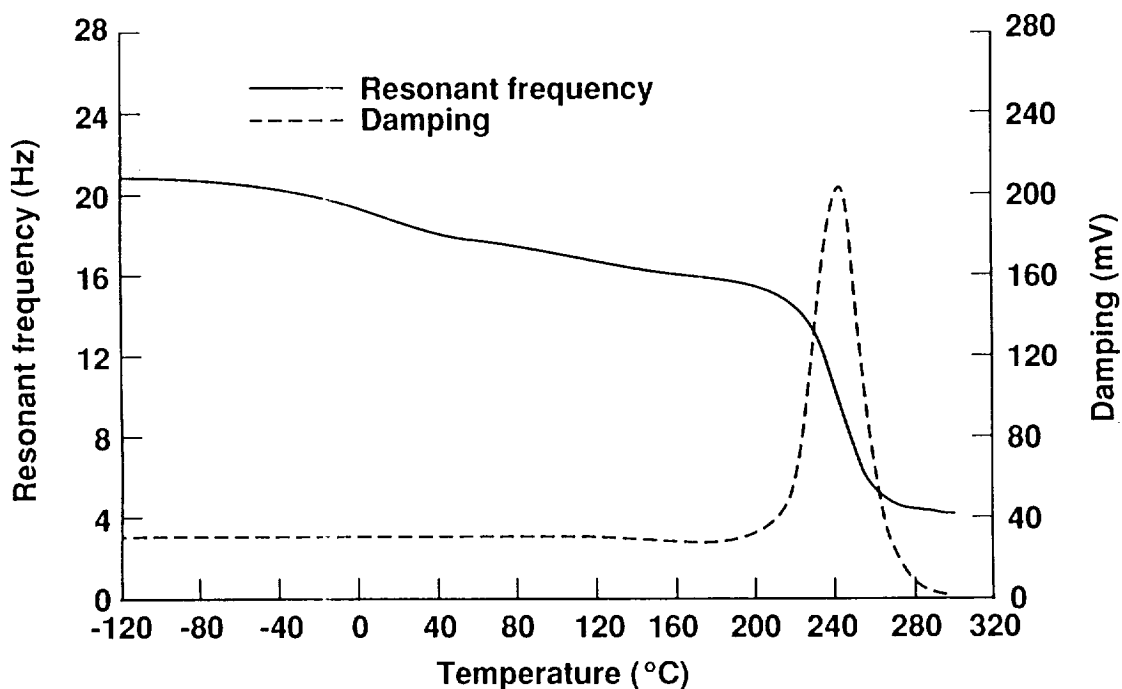


Figure 3. Frequency and damping signal over temperature for a sample of T300/934 graphite/epoxy composite. The resonant frequency decreases in the glass transition region as the stiffness of the sample decreases.

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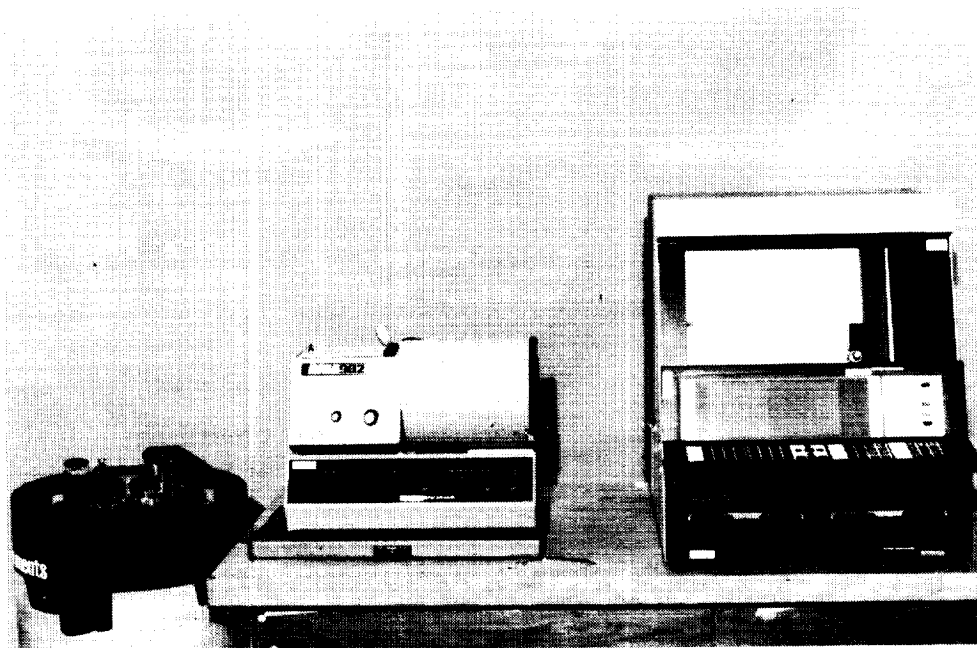


Figure 4. The DuPont 1090 Thermal Analyzer, right, and 982 DMA with heating chamber in place. At the left is the liquid nitrogen tank.

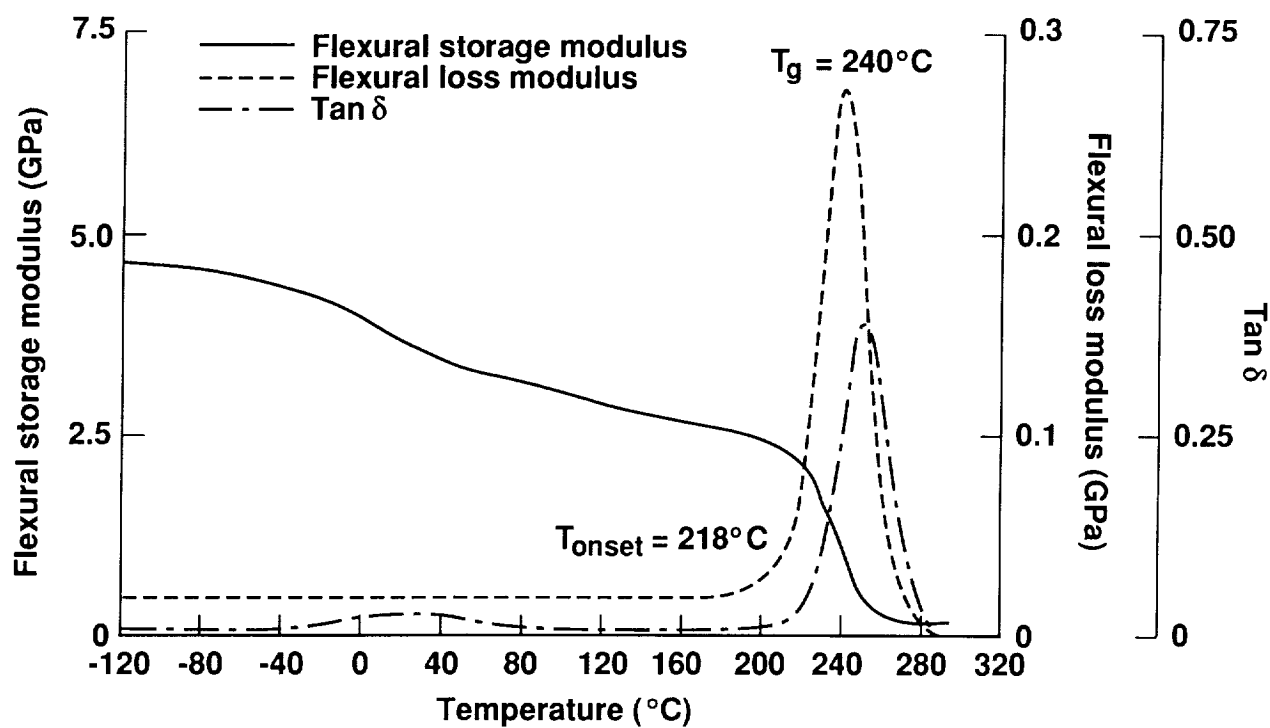


Figure 5. Plots of the dynamic moduli and loss tangent versus temperature calculated from the data on a sample of T300/934 graphite/epoxy composite.

ANODE MATERIALS FOR ELECTROCHEMICAL WASTE DESTRUCTION

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SUMMARY

Electrochemical Oxidation (ECO) offers promise as a low-temperature, atmospheric pressure method for safe destruction of hazardous organic chemical wastes in water. Anode materials tend to suffer severe corrosion in the intensely oxidizing environment of the ECO cell. There is a need for cheaper, more resistant materials. In this experiment, a system is described for testing anode materials, with examples of several common anodes such as stainless steel, graphite, and platinized titanium. The ECO system is simple and safe to operate and the experiment can easily be expanded in scope to study the effects of different solutions, temperatures, and organic materials.

INTRODUCTION

Prerequisite Knowledge Required

The basic experiment can be performed by any technically minded high school student with elementary knowledge of electrical circuits and ionic conduction. The expanded experiment (use of different anolyte solutions, addition of organic "wastes", gas chromatography, etc.) should be performed only by students with a good knowledge of inorganic chemistry and beginning organic chemistry, and an ability to use instrumental methods of analysis. The experiment is based on research being performed at the Pacific Northwest Laboratory to develop ECO as a usable alternative to other waste disposal methods (ref. 1-3).

OBJECTIVES

The objective of the experiment is to demonstrate that any system which is capable of destroying waste materials in an oxidizing environment is itself subject to corrosion, and to find a material which can survive this environment for a long enough time to be commercially useful. Background information on this topic can be found in (ref. 4). After performing the experiment, the student should be encouraged to speculate on possible novel materials which might be more suitable than the expensive platinized titanium (such as conducting plastics, for instance).

EQUIPMENT AND SUPPLIES

Basic Experiment

The necessary equipment comprises:

- A power supply capable of providing up to 6 amps at up to 9 volts.
- Two pumps capable of pumping up to 300 ml/min. of dilute acids or alkalis.
- An Electrochemical Cell (construction described below).
- A sheet of selective anion-permeable membrane (e.g., Riapore 1035).
- Anode and cathode electrodes made of stainless steel, graphite, and a resistant metal such as platinum or platinized titanium.
- Two graduated cylinders (capacity > 500 ml) with a liquid take-off at the bottom (for electrolyte recycle).
- Plastic tubing to interconnect the above.
- A red and a black insulated wire to connect the power supply to the ECO cell.

Expanded Experiment

In addition to the above, the apparatus (shown in Figure 1) can be set up for measuring the effect of temperature, measurement of gases evolved from the anode and cathode compartments, measurement of pH changes during the experiment, etc. For this, additional equipment includes:

- Two hot-plate stirrers to heat the solutions to $<80^{\circ}\text{C}$.
- Tubing take-off from the tops of the graduated cylinders to inverted burettes to measure gas evolution rates.
- A small metering pump for continuous addition of an organic (water-soluble) solvent (in the Figure, the example given is Hexone, or methyl isobutyl ketone).
- Instrumentation as available: Gas chromatograph; pH meter; gas flow meter; gas analyzer (Oxygen and carbon dioxide).

The effect of oxidation conditions on anode materials can be determined visually, or measured with a micrometer gage.

For all experiments, the experimenter has a choice of electrolytes: A dilute acid (e.g., nitric or sulfuric, about 0.5 - 1 M), an alkali (e.g., sodium hydroxide, 1 M), or a salt solution to ensure good ionic conduction.

Cell Construction

ECO cells are available commercially but can be constructed easily for teaching purposes with the facilities of a small workshop. A cell comprises an approximately 7 x 7 cm area. An exploded view of a commercial cell is shown in Figure 2. It is made from laminated teflon sheets, ca. 3 mm thick. Stainless steel end-plates are used to bolt the whole assembly together (4 bolts, one at each corner) tightly enough to prevent leaks. Each electrode plate is held between two plastic plates, with holes drilled at the corners to permit electrolyte flow on both sides of the electrode. The ionic membrane which separates the anode and cathode compartments is held between two sheets of rigid

plastic mesh (some pressure develops within the cell due to osmotic forces). Polypropylene may be used instead of teflon for most purposes, but will not last as long in use. Similarly, connecting tubes between the cell and the pumps and electrolyte reservoirs should preferably be made of teflon, but polypropylene can be substituted. Any design of cell can be used in this experiment, including wires suspended in liquid. Two essential features of any cell used for this experiment are (a) the ionic membrane used to separate the cathode and anode compartments, and (b) recycled electrolytes, or a way of removing gas generated in the cell.

PROCEDURE

An electrochemical flow cell is set up as shown in Figure 1. The flow cell is fitted with a stainless-steel cathode. The anode is made of (a) stainless steel, (b) graphite, or (c) platinum or platinum on titanium. A Riapore 1035 anionic membrane separates the anode and cathode compartments.² The anode and cathode each have an available electrode surface area of 20 cm². The anolytes used in the experiment are generally N NaOH or N nitric acid (total volume of 300 - 500 ml). The catholyte has the same material (acid or alkali), concentration and total volume as the anolyte. If an above room temperature experiment is to be performed, the catholyte and the anolyte are heated and stirred. Burettes inverted over water can be used (optional) to measure gas evolution. Flow of anolyte and catholyte through the cell is started by turning on the two Teflon pumps (Saturn model SP 2000 with Minarik motor controllers). Direct current power is supplied by a Hewlett-Packard model 6281A DC power supply with a maximum capacity of 6 A. Teflon tubing is used to connect all of the system components and Galtek sample valves are used to take samples during system operation.

In a typical experimental run, 500 ml each of anolyte and catholyte are added to the cleaned and leak-checked system. The circulating pumps, heaters, and stirrers are turned on, and the system brought to the desired temperature (this step is omitted for room-temperature experiments). The power supply is then turned on and adjusted to provide 6 A to the cell; the time, voltage, and amperage are noted on the data sheet. The student will note the behavior of the cell, such as visible color changes in the recirculating solutions, which would indicate corrosion of the electrodes. If desired, this can be followed by a simple wet chemistry test or colorimetric measurement for iron (from stainless steel), or carbon dioxide production rate can be monitored to follow the dissolution of a graphite anode.

The experiment can be terminated at any time, preferably after 4-8 hr, and the cell disassembled carefully and cleaned. The degree of corrosion of the electrode can then be determined by direct measurement. As a rule of thumb, a stainless steel anode will show signs of corrosion within 1 hr, from the appearance of a brown insoluble suspension in the anolyte solution; graphite will be completely eaten through in about 6 hr; and platinized titanium will show no effect. Depending on the amount of laboratory time available, each electrode could be subjected to the oxidation conditions in a single experiment (with reassembly of the cell each time), or a single material can be examined in a day. The rate of corrosion can also be adjusted by varying the current through the cell, to suit the instructors convenience.

If an actual organic "waste" degradation is to be performed as well as a measurement of electrode corrosion, a suitable water-soluble compound can be added at a 1-2% concentration before the power is turned on (for safety reasons, to avoid a possible fire if leakage of pure solvent should occur). The anolyte solution is then recirculated for 5 min to mix the organic compound in completely. Hexone (5ml), acetone, diglyme, methanol, or any common solvent can be used. For safety reasons, flammable solvent additions should be monitored or performed by the instructor. Continuous addition can also be performed via a metering pump (as shown in Figure 1). Figures 3 and 4 show typical results of addition and oxidation of common organic substances in the cell.

SAMPLE DATA SHEETS

An example of the data obtained from a typical experiment is shown in Figure 5. The experiment included addition of hexone, and use of a Pt/Ti anode. In this experiment, the anode was inert. Data on the rate of organic destruction is shown in Figure 6.

INSTRUCTOR NOTES

The electrochemical oxidation experiment is widely adaptable in terms of materials studies (corrosion and chemistry), can be adjusted as to time required by simply varying the voltage across the cell, and is simple and safe to perform. Chemicals used are generally dilute acids and alkalies and are relatively nontoxic. Of course, safety glasses should be worn throughout or the experiment performed in a fume hood.

A battery can be substituted for the power supply. Tubing can be either teflon, as recommended, or a cheaper substitute plastic. Obviously, metal tubing will not work. If pumps are not available, a single pass-through, gravity feed of electrolyte can be used.

The electrochemical cell itself could be made in an engineering workshop class, from teflon or polypropylene sheet and stainless steel (for the end plates).

There is unfortunately no real substitute for the ion-permeable membrane or for using some form of platinum as a permanent (non-corrodible) anode. One exercise for the student could be to suggest and perhaps test a potential replacement!

Theory

A simplified theory of electrochemical oxidation is as follows: A hydroxyl ion gives up an electron at the anode, generating a hydroxyl radical ($\text{HO}\cdot$), which can either undergo the normal water electrolysis reaction to form oxygen gas, or can react with an organic compound to hydroxylate it. Enough hydroxylations and even the most resistant organic compound will fall apart to form, eventually, carbon dioxide and water. Of course, hydroxyl radical can also attack the anode and convert a metal into its hydroxide.

Hazards

The experiment itself is remarkably hazard-free. The major problems in practice have been liquid spills of acid, alkali, or solvent, and consequent damage to clothing. Cell leakage has occasionally occurred when an anode plate became perforated and eaten away through the side of the cell. This spillage and leakage problem can be solved by placing a glass oven dish underneath the apparatus to catch spills. Cathode and anode gases should never be mixed, as water electrolysis is a side-reaction and this generates hydrogen (cathode) and oxygen (anode) in explosive proportions. Therefore, the evolved gases should be vented and not allowed near a spark. The pumps and power supply are located outside the hood or remote from the cell for this reason. No smoking should be allowed near the apparatus.

Clean-Up

After the experiment has been completed, power to the cell should be turned off. The electrolytes can then be pumped out, neutralized, and disposed of down the sink (neutralization is necessary to comply with environmental regulations). Used anode materials are non-leachable and can be disposed of directly into the garbage (i.e., graphite, steel, etc.). The cell should be disassembled and thoroughly cleaned after each use: Water is usually sufficient, but acetone or methanol may have to be used to remove iron oxides.

Future Applications

The purpose of this experiment is to expose the student to a developing technology that may one day be of great use for hazardous waste destruction. Electrochemical oxidation is far milder and easier to control than incineration, for example, but it suffers from problems of electrode corrosion. Other problems hampering the wider application of the technology are the fact that the organic waste oxidation takes place only on or near the anode, where hydroxyl (OH.) radicals are generated. Hence a large anode area and/or a high flow rate past the anode are needed. Also, many wastes contain insoluble organics and sludges. Ways to circumvent these problems are needed. In this experiment, the approach is to examine existing engineering materials in the electrochemical oxidation environment and to show that they are either inadequate or too expensive for widespread use. This should encourage the student to suggest alternatives and to start thinking about the problem. Possible topics for discussion are plastic-based conductors and channelized anodes.

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SOURCES OF SUPPLIES

All chemicals required for this experiment are available from most chemical suppliers (e.g., nitric acid, sodium hydroxide, solvents). The rarer electrode materials (platinum on titanium, graphite, nickel, etc.), and the Riapore 1035 ion-permeable membrane can be obtained, for example, from The Electro-Synthesis Co., Inc., P.O. Box 16, E. Amherst, N.Y. 14051. (No endorsement of this particular company is implied; other suppliers may prove equally suitable.) The electrochemical cell can be purchased from this or other supplier of electrochemical equipment, or made in the workshop. Prices are widely variable.

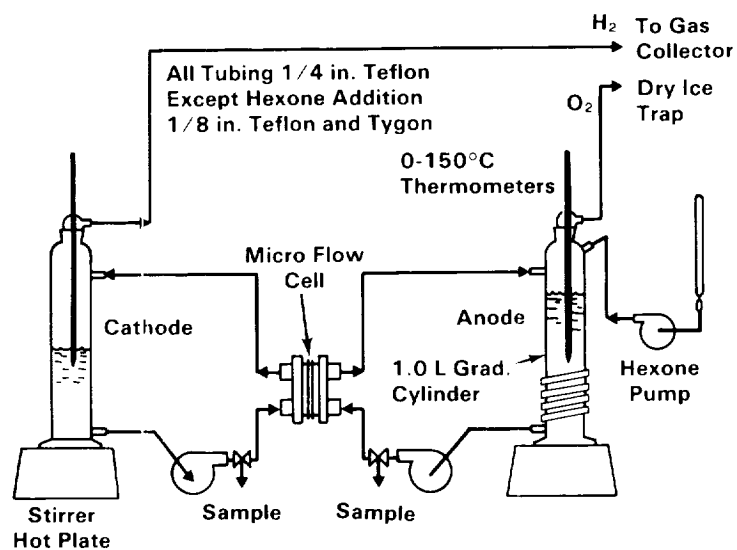


Figure 1: Diagram of Laboratory Apparatus

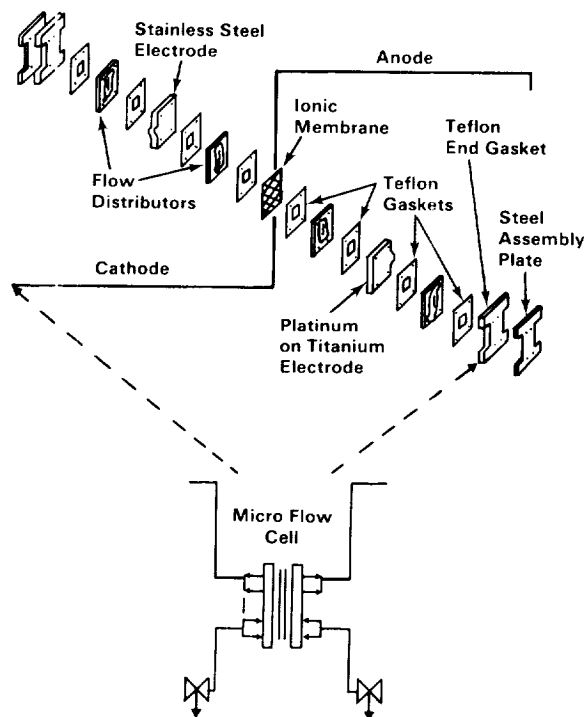


Figure 2: Exploded View of Electrochemical Cell

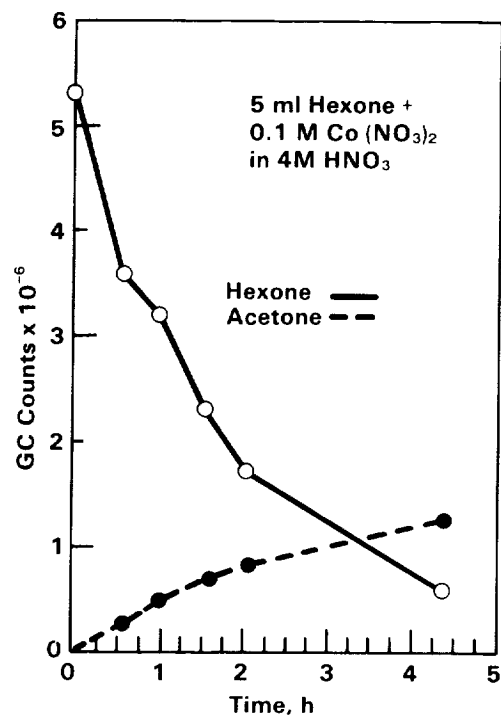


Figure 3: Example of Data Sheet for Hexone Oxidation

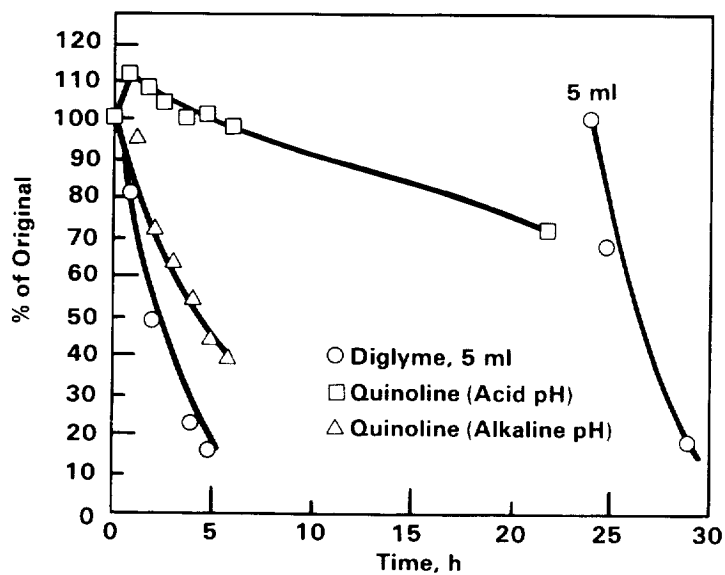


Figure 4: Example of Gas Chromatographic Data for Liquid Sample Analysis

HEXONE DESTRUCTION

TEST # HEX R-38DATE: 8/10/87(stainless steel) CATHODE: 300 cc 4 N nitric acid

PUMP RATES:

(Pt/Ti) ANODE: 300 cc 1 M Co(NO₃)₂ @ 50 cc CATHODE: 300 cc/minin 4 N nitric acid + 5 cc hexone
+ 2.5 cc hexone/hrANODE: 300 cc/min

TIME	POWER		TEMP	ANODE				OBSERVATIONS	CATHODE			
	AMP	VOLTS		GAS SAMPLE	GAS RELEASED	LIQUID SAMPLE	HEXONE COUNTS MIN		TEMP	GAS SAMPLE	LIQUID SAMPLE	OBSERVATION
9:25	60	4.2	47			#1	4.788	0.0241			#2	no cathode off gys
10:07	60	4.1	52			#3	4.855	0.3645				pink tinge in cathode soln
10:48	60	4.2	52			#4	4.128	0.7428				pink-purple tinge
11:04	60	4.2	52		635 cc			off gys are seen to be increasing				
11:25	60	4.2	50			#5	3.725	0.9096				deep pink-purple color
11:50	60	4.2	50		increasing loss cc							
12:24	60	4.2	50		635 cc	#6	3.300	1.183				
13:10	60	4.2	51		increasing loss cc	#7	2.975	1.242				
13:43	60	4.2	50		635 cc							
13:52	60	4.3	50			#8	2.473	1.115				
14:11					635 cc							
14:35	60	4.4	50			#9	2.533	1.086				
14:45					635 cc							
3:05					635 cc							
3:09	60	4.4	50		635 cc	#10	2.167	0.894	trace of DOX			brassy purple soln
3:48	60	4.5	50		635 cc	#11	1.664	0.6084	trace DOX		#11	brassy purple soln

Figure 5: Example of Graphical Data Presentation for Hexone Oxidation

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OF POOR QUALITY

HEXONE DESTRUCTION DATA ANALYSIS:

Gas Chromatograph Standard

% hexone	GC counts	slope	intercept
1	3566000	2.821E-07	-0.0059238
0.1	375500		

Date --> June 4, 87		Hexone Increment Destruction								
Elap. Min.	Time	Interval	Hexone GC Counts	gas Hexone Remaining	gas Hexone Destroyed	gas Hexone Destroyed	Rate gas/hr	Amps	Volts	
-0	11:16		3290000	5.00				6	4.9	
30	11:46	30.0	2546000	3.87	1.13	1.13	2.2614	6	4.9	
60	12:16	30.0	1862000	2.83	2.17	1.04	2.0790	6	4.8	
90	12:46	30.0	1515000	2.30	2.70	0.53	1.0547	6	4.9	
180	14:16	90.0	390800	0.59	4.41	1.71	1.1390	6	4.9	
210	14:46	30.0	112800	0.17	4.83	0.42	0.8450	6	5	
240	15:16	30.0	23490	0.04	4.96	0.14	0.2715	6	5	

June 5, 87		Hexone Increment Destruction								
Elap. Min.	Time	Interval	Hexone GC Counts	gas Hexone Remaining	gas Hexone Destroyed	gas Hexone Destroyed	Rate gas/hr	Amps	Volts	
0	10:15		5228000	5.00				6	4.2	
32	10:47	32.0	3870000	3.70	1.30	1.30	2.4352	6	4.2	
90	11:45	58.0	1731000	1.66	3.34	2.05	2.1163	6	4.3	
154	12:49	64.0	905800	0.87	4.13	0.79	0.7399	6	4.4	
203	13:38	49.0	293000	0.28	4.72	0.59	0.7176	6	4.5	
269	14:44	66.0	33960	0.03	4.97	0.25	0.2252	6	4.5	

Figure 6: Example Data Presentation for Diglyme and Quinoline Oxidation

A SIMPLE DEMONSTRATION OF CORROSION CELLS

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Key Words: Corrosion, Galvanic Cell, Polarization

Prerequisite Knowledge: Concurrent with classroom lectures on corrosion theory and phenomena, subsequent to laboratory and classroom discussions on cold working and the microstructural heterogeneity of most metals.

Objective: Reinforce and enhance the understanding of galvanic cells, anode and cathode reactions and polarization phenomena.

Equipment and Supplies:

- (8) Common Nails, at least 16d size (at least 90 mm long)
- (1) Galvanized Nail, same size (Alternatively, another mild steel nail and a strip of zinc can be substituted.)
- Bare copper wire or strip
- Tin wire or strip, alternatively lead wire or strip
- (9) Glass test tubes with rubber stoppers
- Test Tube Rack
- Distilled water
- Tap Water

Procedure:

The instructor should put the following materials into test tubes two days in advance of the laboratory session:

- Tube 1 Clean steel nail covered with tap water
- Tube 2 Clean steel nail covered with tap water, boiled to drive off dissolved oxygen
- Tube 3 Clean steel nail covered with distilled water, boiled to drive off dissolved oxygen
- Tube 4 Steel nail, as received, covered with tap water
- Tube 5 Clean steel nail partially immersed in tap water
- Tube 6 Galvanized nail, one side ground flat, (alternatively, a clean steel nail, wrapped with a narrow (3-5mm) strip of zinc coiled into a helix) covered with tap water
- Tube 7 Clean steel nail, wrapped with copper wire or strip, covered with tap water
- Tube 8 Clean steel nail, wrapped with tin wire or strip, covered with tap water
- Tube 9 Clean steel nail

All tubes should be stoppered, placed in a test tube rack, and left undisturbed.

During the laboratory session, students should inspect each of the test tubes (CAREFULLY -- DO NOT SHAKE OR JAR THE TEST TUBES IN ANY WAY!). Observations should be noted regarding the appearance of the materials in each of the tubes.

Students' reports for this laboratory session should contain all of the following elements:

Summary

Description of Experiment

A sketch of the appearance of the nail in each test tube with all elements carefully and completely labeled.

Analysis of Data

For each test tube, the student should identify:

- any evidence suggesting that a galvanic cell is operating,
- the anode and cathode regions,
- the most likely chemical reactions occurring at the anode and cathode.

Conclusion

A concise restatement of the necessary conditions for galvanic corrosion and the bases for anodic and cathodic polarization.

Remarks regarding which of the cells were most informative in supporting the theory of corrosion discussed in classroom lectures.

Interpretation of Corrosion Cells:

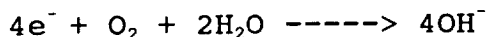
Tube 1 Galvanic corrosion occurs when two materials of different solution potential are electrically connected and situated within an electrolyte. For the nail in this test tube, the presence of rust on the head, the point, and, less distinctly, on the ridges under the head along with the absence of rust on the shank are evidence of a galvanic reaction. The tap water is the electrolyte. The rusted areas, being more severely cold worked than the shank, are anodic regions. The shank is the cathode.

Tube 2 All of the elements needed for corrosion are present--anode, cathode and electrolyte. However, there is little evidence of rust because the water does not contain a large quantity of the dissolved oxygen necessary to form $\text{Fe}(\text{OH})_2$. The anode and cathode areas are the same as in Tube 1.

- Tube 3 This cell exhibits even less reaction than Tube 2. Because the distilled water is deficient in ions, it is a poorer electrolyte than tap water. The galvanic reaction is suppressed.
- Tube 4 The extent to which less rust is present here than in Tube 1 depends on the amount of grease and wax remaining on the surface from the nail forming process. This coating temporarily inhibits the current flow necessary for corrosion. Oxygen concentration cells quickly form at scratches in the coating, separating it from the metal. Soon the galvanic action becomes more uniform, and the appearance becomes similar to that of Tube 1. A galvanic cell is operating.
- Tube 5 The nail has a dense ring of rust at the waterline. Close observation will show pitting attack of the steel just below the waterline. The corrosion cell results from the difference in the oxygen concentration at the water surface and the concentration at some depth. The cathode region occurs at the surface and the anode region is just under the surface.
- Tube 6 The exposed steel on the galvanized nail is covered with bubbles. Of the metals present, the zinc is the anode, and the exposed steel is the cathode. The bubbles are hydrogen gas produced by the cathode reaction:
- $$2e^- + 2H_2O \rightarrow H_2 + 2OH^-$$
- The hydrogen gas bubbles are evidence of activation polarization at the cathode which is slowing the rate of corrosion by forming a physical barrier to further reaction. A white precipitate, zinc oxide or hydroxide, may be visible at the bottom of the tube, further evidence that the zinc is undergoing oxidation. Of the metals used in this demonstration, zinc is the only one which is sufficiently anodic to displace hydrogen from water.
- Tube 7 The nail is covered with the same pattern of rust as in Tube 1. The anodic areas are the head, the point and the ridges; the cathodic areas are the copper wire and, to some extent, the shank. The copper affords no protection against corrosion. In fact, it accelerates the corrosion reaction because the difference in galvanic potential between the copper and steel is greater than that between the two regions of steel having different degrees of cold work, as was the case with Tube 1.
- Tube 8 The appearance is essentially identical to Tube 7. Even though tin is slightly more anodic than copper, it is cathodic to steel. There is no visual evidence of polarization. (If lead is substituted for the tin, the same comments apply.)
- Tube 9 A galvanic cell is not operating because one of the necessary conditions is not met; an electrolyte is not present.

Notes for the Instructor:

The use of common nails to demonstrate the principles of corrosion has been described by several authors¹⁻³. The experiment described in two of the references is particularly interesting in that color indicators are added to the electrolyte to delineate the anodic and cathodic reactions. Phenolphthalein is used to indicate by the formation of a pink color the presence of hydroxyl ions formed by the cathode reaction:



Potassium ferricyanide, $K_3Fe(CN)_6$, indicates the presence of ferrous ions by the formation of the dark blue compound, ferrous ferricyanide. The electrolyte employed is an agar gel which keeps the reaction products near their points of formation. The reactions can even be carried out in a flat-bottomed glass dish placed on an overhead projector. However, the preparation time for such a demonstration is more extensive because of the time needed to prepare the agar-based electrolyte.

We have found that our abbreviated version of the common nail corrosion experiment is elegantly simple to prepare, yet pedagogically efficient in that it directs students to make observations that focus on the elements of galvanic cells and basic polarization phenomena.

If a few simple precautions are taken, the corrosion cells will react to a stage at which the products, either rust or hydrogen bubbles, are easy to observe and interpret.

Buy big nails. This precaution ensures that the point and head are heavily cold worked.

Clean the surfaces of the nails with 220 grit silicon carbide sandpaper and wash in methanol. Alternatively, clean the surfaces by glass bead blasting. Metal wire or strip used to wrap the nail should also be lightly abraded to remove any surface film which might interfere with good electrical contact. Reserve one nail in its as-received condition for Tube 4.

When nails are wrapped with a strip of another metal, the helix should be tight enough to ensure adequate electrical contact between the two metals. However, ample space should be left between neighboring coils, so that the surface of the nail is readily visible.

If the test tubes are of a heat-resistant glass, the water can be boiled directly in Tubes 2 and 3 with the nails in place. The tube should be stoppered immediately to prevent contact with the atmosphere.

If lead or tin strip is unavailable, it can be made by pouring a small melt onto a steel block, hammering the solidified metal to a thickness of about 1.0 mm and then cutting off ribbons with tin snips. If a small rolling mill is available, especially one with grooves for making wire, the task is much easier. Be sure to skim the dross off from the melt just before pouring.

The galvanized nail must have one side ground flat to remove the zinc coating and to expose the steel. Use a pedestal grinder and grind deeply into the nail so that a large area of the steel is exposed. When the grinding is finished, clean the ground galvanized nail with 220 grit sandpaper and methanol.

When all of the test tubes are filled and placed in the rack, set it as close to eye level as possible, and post a fearsome sign prohibiting any disturbance. During the laboratory session, encourage students to rotate the tubes gently and inspect all surfaces. The corrosion products are surprisingly secure; with some care they can survive being passed around a classroom.

In those cells containing fresh tap water it should be noted that, as cold tap water warms up to room temperature, dissolved air may come out of solution and form small bubbles on the walls of the test tube and on the metal specimens. These bubbles may be dislodged, if desired, by gently tapping the tubes a few hours after the experiment is set up. Alternatively, the tubes may be left undisturbed so that the students can compare them to those tubes containing boiled water. The air bubbles should not be confused with the hydrogen bubbles which form on the exposed steel of the galvanized nail. The latter are generally much bigger than the former and will re-form repeatedly if they are dislodged.

After the laboratory session, discard the water and save all of the test tubes, nails, tin and lead strips and copper wire for the next semester's class. Rust deposits left too long are difficult to remove.

We have found that this demonstration graphically and easily illustrates the basic principles of corrosion. The following facts are clearly demonstrated.

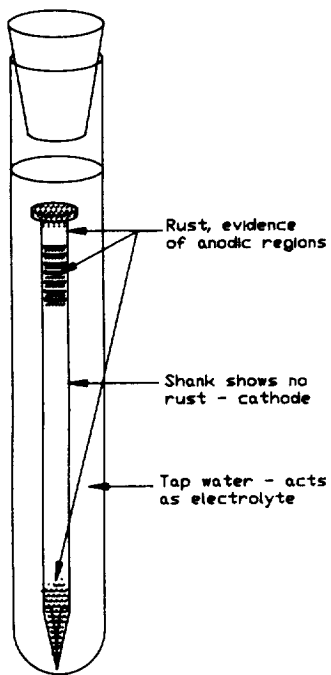
Galvanic corrosion cells do not require the presence of two different metals. Regions having the same chemical composition but differing degrees of cold work are sufficiently different in chemical reactivity that an anode and cathode can occur in two regions on the same object.

The presence of two different metals promotes corrosion. Steel can be either the cathode or the anode depending on what second metal is present.

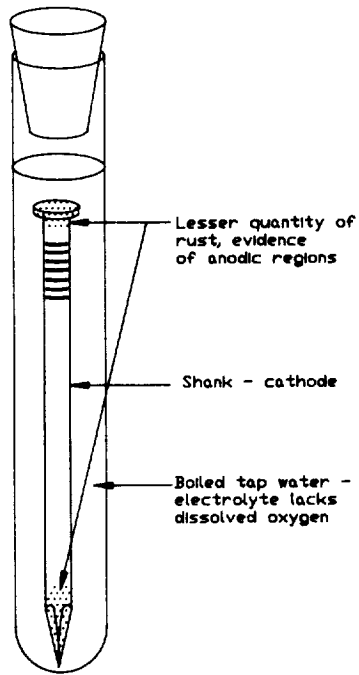
An electrolyte is necessary for corrosion.

Once materials for this demonstration are gathered, they are conveniently stored and reconstituted in subsequent academic sessions. Resurrecting the demonstration requires a brief cleaning of the surfaces and the addition of water. The ease with which this experiment can be set up using readily available materials makes it a valuable addition to the metallurgical teaching laboratory.

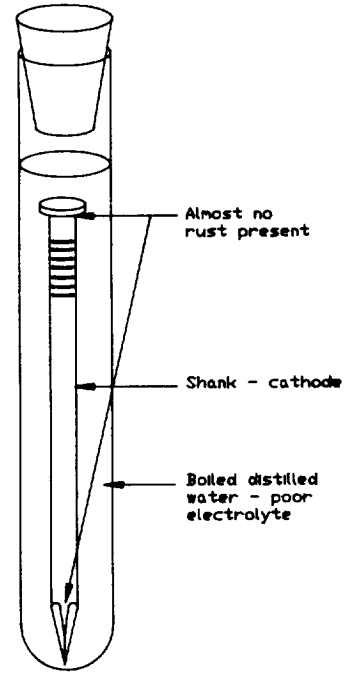
1. Corrosion in Action, The International Nickel Company, New York, 1977, pp. 15-18.
2. D. Eurof Davies, Practical Experimental Metallurgy, Elsevier, New York, 1966, pp. 118-122.
3. B.R. Schlenker, Introduction to Materials Science, Wiley, New York, 1969, p. 68.



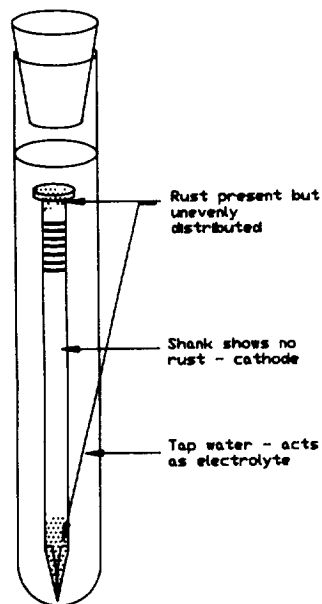
Tube 1



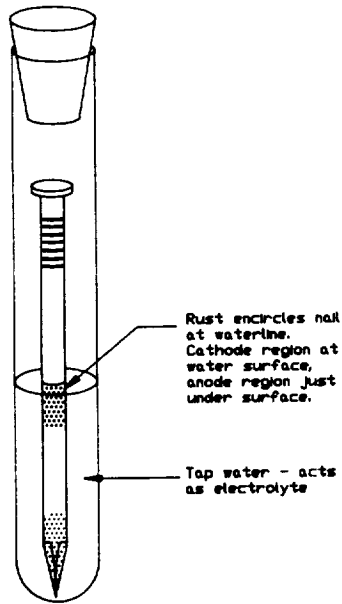
Tube 2



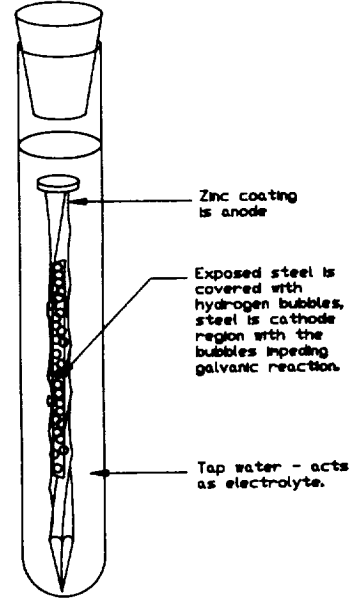
Tube 3



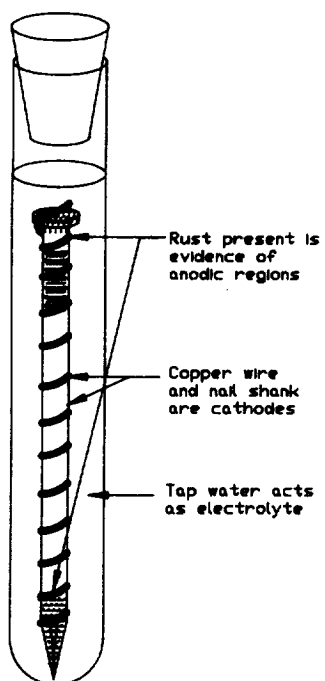
Tube 4



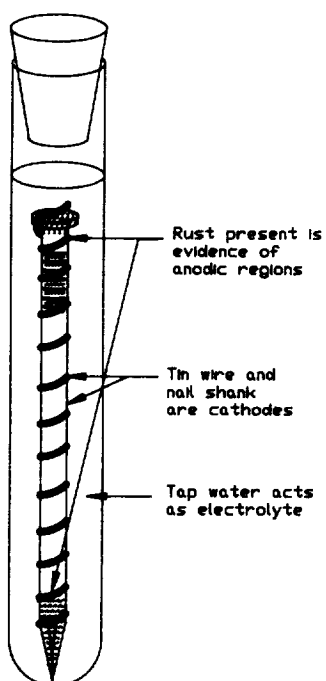
Tube 5



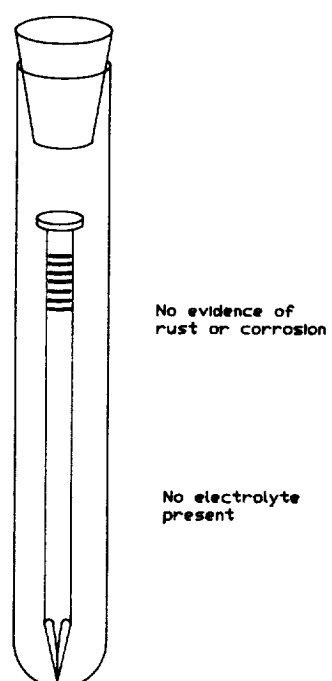
Tube 6



Tube 7



Tube 8



Tube 9

THERMAL CONDUCTIVITY OF METALS

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OBJECTIVE

To familiarize students with steady and unsteady heat transfer by conduction and with the effect of thermal conductivity upon temperature distribution through a homogeneous substance.

Prerequisite or corequisite:

- a) Physics - high school or college freshman course, with heat topic which introduces conductivity property and units.
- b) Introductory discussion of concept of heat transfer in the laboratory, including a handout to give students an appropriate background.

INTRODUCTION

A knowledge of material structure and properties is a prerequisite for the selection and safe use of engineering materials by the designer. Structure, generally studied and postulated by scientists, determines properties which characterize the behavior of interacting materials, exposed to different effects. For a designer, strength and hardness are the important mechanical properties for a part to be loaded or indented by outside force. Moreover, the designer should also consider thermal properties, for parts to serve at temperatures other than as fabricated, or to perform some heat-transfer function.

For example, when a metal rod is hot worked (or heated) it exhibits three thermal effects: a) the rod absorbs heat; b) it expands; c) it transmits heat. Absorption of heat is characterized by the property "specific heat capacity" c_p ; i.e., the energy Q (Joules) required to raise one unit mass m (kg) by temperature change ΔT of one degree (K°). Expansion is usually described by "coefficient of linear thermal expansion" (α linear expansivity); i.e., fractional change in original length, $\Delta L/L$ per unit change in temperature ΔT (K°). Heat transmission is identified mainly by thermal conductivity (k), a measure of the ease of thermal-energy transmission through a body; i.e., the rate of energy (Joules/s) in a unit thickness Δx (m) of unit area A (m^2), and unit change in temperature ΔT (K°).

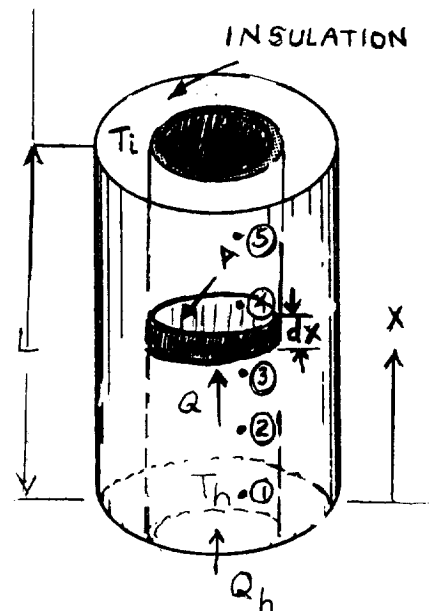
Thermal conduction is important in modern manufacturing processes: Heat treating, die casting, plastics molding, heat-sink soldering and platen heating. The two other forms of heat

transmission (convection and radiation) may also be present but their effects are often negligible. Hence, an intuitive understanding of heat conduction is essential for materials engineers. Materials textbooks, handbooks, and standards (e.g., ASM Metals Handbook, CRC Physics and Chemistry Handbook, CINDAS database) give exhaustive tables of thermal properties of a myriad of engineering materials, but rarely do references describe an experiment to measure thermal conductivity.

This paper presents a simple heat conduction experiment to give insight into the concept of steady and transient heat transfer. Students can measure the temperature distribution along the heat path, learning how thermal conductivity differs for different materials. After presenting the theoretical backgrounds, test equipment and procedure will be covered, data sheet exhibited, sample calculations outlined, graphs plotted and some concluding remarks made.

THEORY

Heat flows naturally from a high-temperature point to a low-temperature point. Consider a homogeneous metal rod (mass m , cross-section A , length L) with perfectly insulated perimeter and five equally spaced thermocouples embedded to the center of the rod (Figure 1). Assume the rod is initially at temperature T_i . If the upper face stays at temperature T_i , while the lower face is suddenly subjected to a higher constant temperature T_h heat source, the molecules on that face start absorbing thermal energy, vibrating at higher velocity and passing some of their energy to adjacent molecules in the metal. Furthermore, "free electrons" start to drift and transfer "heat" along the rod.



As time passes, some of the "thermal energy" flows continuously in the x -direction. Some of the thermal energy is partially stored (absorbed) in the bar, continuously increasing its "temperature" along the bar; and if imposed conditions on the faces are maintained, heat flows up the rod, and temperature at each part will always be lower than a preceding point.

Now, consider a disk element (thickness dx) across which the temperature change is dT and the heat flow-rate is Q . According to Fourier's law of heat conduction, the amount of heat transferred is

$$Q = - kA (dT/dx) \quad (1)$$

where, dT/dx is "temperature gradient", the temperature drop per unit length; k is "thermal conductivity" of the material. The negative sign indicates heat flowing towards decreasing temperature.

The rate of energy stored dE in the element is given by

$$dE = m c_p dT/dt, \quad (2)$$

where, c_p is "specific heat capacity", and t is "time".

The condition where the temperature T at a position (x) is continuously changing with time, is called unsteady (transient) state.

If imposed conditions are maintained long enough, there comes a time after which the temperature at any given point does not change with time, no more energy is being stored in the rod; hence the rate of heat entering the lower face will equal the rate leaving the upper face, or $dE = 0$. This is called "steady state". For a homogeneous substance with constant thermal conductivity the steady state rate of heat flow (Q) could be written as

$$Q = - kA \Delta T/\Delta x \quad (3)$$

where Δ refers to finite change.

Consider several different metal rods with the same dimensions and the same steady state Q . If k of one sample is known, one can determine k for any other sample: Let the samples be numbered 1,2,3...

$$\text{Then, } Q = - k_1 A (\Delta T/\Delta x)_1 \quad (4)$$

$$Q = - k_2 A (\Delta T/\Delta x)_2 \quad (5)$$

$$\text{Hence, } \frac{k_2}{k_1} = \frac{(\Delta T/\Delta x)_1}{(\Delta T/\Delta x)_2} \quad (6)$$

Temperature gradient ($\Delta T/\Delta x$) is found by plotting measurements of T vs x , which also gives the "temperature distribution" showing temperature at any point on the bar.

Good electrical conductors are good thermal conductors, as indicated by the "Wiedemann-Franz" relationship, for a given temperature: k is proportional to σ , or

$$k = L T \sigma \quad (7)$$

where, L is a published constant.

For two different metals at the same temperature, using the above relationship, one may write

$$k_2/k_1 = \sigma_2/\sigma_1 \quad (8)$$

If the two σ or their ratio σ_2/σ_1 and one thermal conductivity are known or estimated, the other thermal conductivity can be evaluated.

EXPERIMENTATION

A. Test Equipment

The specimens: aluminum alloy (6061 T6), brass (yellow), and steel (cold rolled) had thermocouples connected at distances of 0.5, 3.0, 5.5, 8.0, and 10.5 cm from the bottom of the specimens which were wired into an Omega Trendicator that reads temperatures at each point.

The specimens were mounted in a bucket and surrounded with insulation leaving only the top of the specimen exposed and the bottom touching the bucket. The bucket was then placed on a Thermolyne Corporation hot plate which was preheated to 200°C. A stopwatch was used for timing the readings.

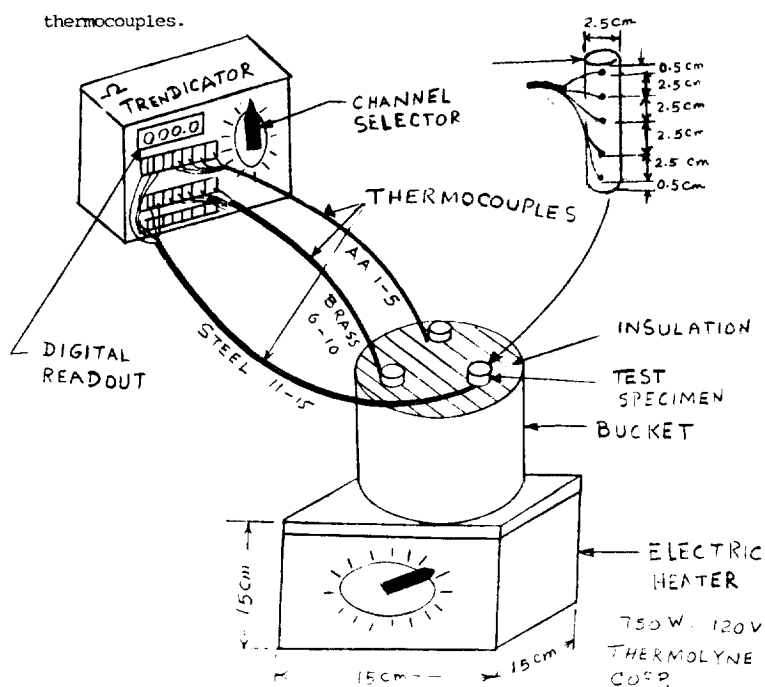


FIGURE 1

B. TEST PROCEDURE

The heater (hot plate) was turned on, set at about the 400 watt mark (~200°C) and left to achieve steady state. Then, while the bucket of specimens was placed on the hot plate, the stopwatch was started. The first 5 runs of temperatures vs time were read after every 2 minutes and afterwards every 5 minutes till steady state was achieved or approached. Then, the hot plate and thermocouple indicator were turned off. The whole experiment took 60 minutes.

MET 220 LAB

DIV. _____

ROOM TEMP. _____

BAR. PRESS. _____

DATA SHEET

EXPERIMENT 6: HEAT CONDUCTION

NAME _____

COWORKERS _____

DATE _____

TIME _____

Run No.	Time Min.	TEMPERATURE °C														
		ALUMINUM ALLOY					BRASS					STEEL				
1	0	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2
2	2	48.0	40.1	37.8	35.8	35.9	52.8	51.7	32.0	29.8	28.1	54.7	36.8	30.3	25.4	24.0
3	4	60.0	61.9	48.9	47.8	47.2	65.0	51.8	46.9	40.4	38.2	68.3	48.3	40.3	35.7	31.1
4	6	73.1	65.0	61.7	59.3	60.5	77.8	64.7	59.2	53.3	49.5	79.0	58.3	50.7	44.6	40.2
5	8	81.7	73.2	71.1	68.3		86.7	73	68.3	63.2	59.7	88.9	67.8	60	55.4	49.3
6	10	91.8	84.1	82.2	78.3	79.7	96.0	84.4	78.3	73.8	68.4	97.2	76.7	67.4	62.9	58.
7	15	110.5	104.1	101.1	98.2	99.4	116.1	105	100.	94.4	86.7	115.6	96.7	88.9	83.6	77.3
8	20	125	118.9	115.3	112.7	113.6	130	118	115.2	110	102.2	129.8	115.7	106	100	92.6
9	25	137.8	132.3	129.1	125.5	120.7	142	133.6	128.8	123.7	118.9	130.3	129.8	120.3	114.4	106
10	30	145.4	140.	136.6	133.4	133.8	153.8	141.6	136.2	130.6	121.1	145.7	141.5	132.1	124.2	112.4
11	35	151	146.7	143	140.1	140	155	146.1	143	138.8	127	155	151.6	137.8	131.2	123.6
12	40	154.4	152.2	148.7	145	144	159.3	153.3	149	143	130.9	157.8	152.1	143.3	138.4	129.3
13	45	160	155.5	152.8	148.9	148.2	163.3	157.2	152.1	147	134	163.7	158.7	148.4	143.3	132.3
14	50	163.1	158.7	156.8	151.8	150.9	166	160.6		150.3	150.7	166.4	162.4	152.5	146.4	136.8
15	55	165.8	160.	157.2	153.4	152.7	167.2	162.1	159.4	153.3	157.4	168.1	162.4	155.2	149.6	138.9
16	60	166.4	161.4	158.3	155.2	153.7	168.8	163.8	158.7	153.7	159.5	169	164.4	157.2	152	140.7

FIGURE 2

EVALUATIONS:

1. Graph temperature T versus time t for the middle point of each specimen on the same sheet of graph paper. Label graphs completely and identify the transient and steady state regions.

Answer: See Figure 3

2. On the same sheet, graph the temperature versus distance (position) along the bar for the steady state (last set of readings), and determine the temperature gradient for each sample.

Answer: See Figure 4 for the graph

$$(\Delta T / \Delta x)_{AA} = -1.133 \text{ } ^\circ\text{C/cm} = -113.3 \text{ } ^\circ\text{C/m}$$

$$(\Delta T / \Delta x)_{Br} = -1.808 \text{ } ^\circ\text{C/cm} = -180.8 \text{ } ^\circ\text{C/m}$$

$$(\Delta T / \Delta x)_{St} = -2.33 \text{ } ^\circ\text{C/cm} = -233 \text{ } ^\circ\text{C/m}$$

3. Estimate the thermal conductivity ratios k_{Br}/k_{AA} k_{St}/k_{AA}

Answer:

$$\text{From Equation (6)} \quad k_{Br}/k_{AA} = \frac{(\Delta T / \Delta x)_{AA}}{(\Delta T / \Delta x)_{Br}} = \frac{-1.133 \text{ } ^\circ\text{C/cm}}{-1.808 \text{ } ^\circ\text{C/cm}} = 0.627$$

$$k_{St}/k_{AA} = \frac{(\Delta T / \Delta x)_{AA}}{(\Delta T / \Delta x)_{St}} = \frac{-1.133 \text{ } ^\circ\text{C/cm}}{-2.33 \text{ } ^\circ\text{C/cm}} = 0.486$$

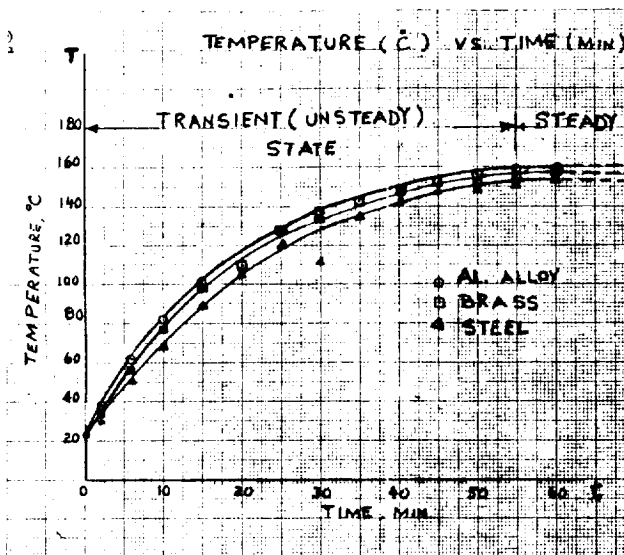


FIGURE 3 ↑

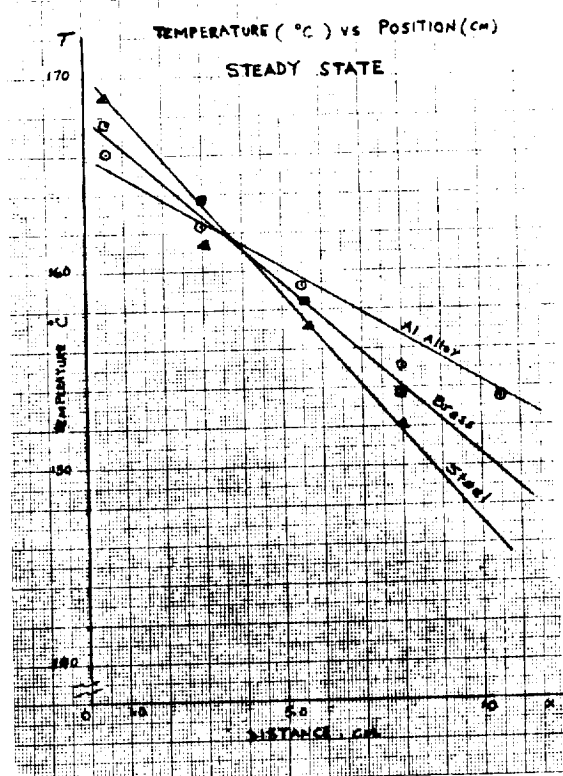


FIGURE 4 →

4. Compare the k ratios found experimentally with the true ratios.

Answer: From the references $k_{AA} = 168.1 \text{ J/s mK}^\circ$
 $k_{Br} = 98.8 \text{ J/sm K}$ $k_{St} = 72.8 \text{ J/s mk}$

Therefore,

$$(k_{Br}/k_{AA}) \text{ true} = 0.587 \quad (k_{Br}/k_{AA}) \text{ exp} = 0.627; \quad \therefore \text{error} = 6.7$$

$$(k_{St}/k_{AA}) \text{ true} = 0.433, \quad (k_{St}/k_{AA}) \text{ exp} = 0.486; \quad \therefore \text{error} = 12.2$$

5. From the references find the thermal conductivity for each specimen, determine the σ ratios and compare them with the true k ratios.

Answer:

$$\begin{array}{llll} \sigma_{AA} = & 0.222 & \Omega^{-1} & \text{cm}^{-1} \\ \sigma_{Br} = & 0.158 & \Omega^{-1} & \text{cm}^{-1} \\ \sigma_{St} = & 0.034 & \Omega^{-1} & \text{cm}^{-1} \end{array}$$

$$\frac{\sigma_{Br}}{\sigma_{AA}} = 0.712 \quad ; \quad \frac{\sigma_{St}}{\sigma_{AA}} = 0.150$$

6. Calculate Q/A for each sample using the true k value and $\Delta T/\Delta x$

Answer:

$$(Q/A)_{AA} = -k_{AA} (\Delta T/\Delta x)_{AA} = 19046 \text{ J/sm}^2$$

$$(Q/A)_{Br} = 17,863 \text{ J/s m}^2 \quad , \quad (Q/A)_S = 16,962 \text{ J/sm}^2$$

REMARKS

The elementary heat conduction experiment presented is designed, for associate degree technology students, in a simple manner to enhance their intuition and to clarify many confusing concepts such as temperature, thermal energy, thermal conductivity, heat, transient and steady flows. The equipment set is safe, small, portable (10 kg) and relatively cheap (about \$1200): The electric hot plate 2 kg (4.4 lb) for \$175; the 24 channel selector and Thermocouple Digital Readout (Trendicator) 4.5 kg (10 lb) for about \$1000; the three metal specimens (each of 2.5 cm diameter and 11 cm length), base plate and the bucket all about 3 kg (7 lb) for about \$25. The experiment may take from 60 to 70 minutes. Although the hot plate surface temperature could be set from 90 to 370 °C (maximum of 750 watts) it is a good practice to work with temperatures of 180 to 200 C (about 400 watts). Students should be reminded of safety when working with high temperatures and voltages. They may experiment in squads of 2, 3 or even 4, or the instructor may demonstrate it for the whole class.

One part of exercises requires that the students use references for finding the true k and σ values. It is a challenge.

SYMBOLS

AA	Aluminum Alloy
Br	Brass
ASM	American Society of Metals
ASTM	American Society for Testing Materials
CINDAS	Center for Information and Numerical Data Analysis and Synthesis
J	Joule(s)
k	kappa (thermal conductivity), $J^{cm}/s\ cm^2C^{\circ}$ or W^{cm}/cm^2C°
K°	- Kelvin degree (temperature difference)
K	- Degrees Kelvin (temperature at a point)
m	- metre, meter
S	- sec, seconds
st	steel
W	- watt(s)
Ω	(omega) - Ohm.

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THE ASSESSMENT OF METAL FIBER REINFORCED POLYMERIC COMPOSITES

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ABSTRACT

Because of their low cost, excellent electrical conductivity, high specific strength (strength/density), and high specific modulus (modulus/density) short metal fiber reinforced composites have enjoyed a widespread use in many critical applications such as automotive industry, aircraft manufacturing, national defense, and space technology. However, little data has been found in the study of short metal fibrous composites. Optimum fiber concentration in a resin matrix and fiber aspect ratio (length-to-diameter ratio) are often not available to a user. Stress concentration at short fiber ends is the other concern when the composite is applying to a load-bearing application. Fracture in such composites where the damage will be initiated or accumulated is usually difficult to be determined. An experimental investigation is therefore carefully designed and undertaken to systematically evaluate the mechanical properties as well as electrical properties. In this study, Inconel 601 (nickel based) metal fiber with a diameter of eight microns will be used to reinforce commercially available thermoset polyester resin. Mechanical testing such as tensile, impact, and flexure tests along with electrical conductivity measurements will be conducted to study the feasibility of using such composites. The advantages and limitations of applying chopped metal fiber reinforced polymeric composites will also be discussed.

INTRODUCTION

Over the years, polymers have been well known for their electrical insulating properties and great strides have been made in electrical and electronic applications, mainly related to electrical insulation. Consequently, research has been directed to improve the dielectric strength of polymers so that they can be used for better insulators. In the past ten years, with the advent of electrically conductive polymers, their potential to perform as active roles in conducting electricity has been discovered and realized (ref. 1). Recent polymer researches have revealed that polymers can indeed conduct electricity as well as metals. Now the electrically conductive polymers can be used as antistatic coatings, fuel cell catalysts, solar electrical cells, photoelectrodes in a photogalvanic cell, protective coatings on electrodes in photoelectro-chemical cells, and as lightweight, inexpensive batteries.

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Add to this developing need for electrophotographic industry, due to the increasing need of lightweight, low cost, moldable, and high specific strength for defense and high tech applications, it is expected that the development of electrically conductive polymeric materials will grow strongly and significantly. On the other hand, conductive plastic housings and molded parts can also be beneficial to the controls of electromagnetic interference shielding (EMI) and electrostatic charge discharge (ESD). Advanced research studies have shown that there are three possible methods to make polymers conductive. The first approach is to apply a thin conductive coating onto the molded part. This approach, however, is costly and not efficient because of involving a two-step operation which increases the difficulties in obtaining a good adhesion as well as a uniform coating. The second approach is often held by synthesis or by doping (ref. 2-5). Synthesis is done by side reactions. One of the major side reactions involves the benzene ring. Other reactions lead to branched and cross-linked polymers. Doping involves oxidation and reduction reactions. This method, usually produces polymeric compounds such as polyacetylene and polyphenylene, although it has been proven to be effective and has been widely used, problems arise from conductive polymers themselves such as their processability, stability, mechanical and physical properties, etc. The last approach, proposed in this study, is to incorporate electrically conductive fillers in the polymeric resin matrix. Many conductive materials such as carbon, metals, metal-coated fillers in the form of particles, particulates, and fibers can be randomly dispersed into a resin matrix and form a so-called "conductive composite." This approach so far appears to be a viable solution to the development of conductive polymers. Due to the lack of systematic research study in this area, limited data can be found to help research scientists, engineers in industries in the application of these materials. More importantly, much research is urgently needed to fully understand the interrelationships among structure, property and processing prior to their commercial utilizations (ref. 1-6).

The conductive polymeric composite was first presented in 1966 by Garland (ref. 7). He used silver particles, approximately 50 to 200 microns in diameter, to reinforce a thermoset phenol-formaldehyde (Bakelite) resin matrix. His experimental data indicated that metal-filled polymers undergo a sharp transition from an insulator to a conductor at a critical volume concentration of metal fillers. In his study the electrical resistivity remained almost constant until the silver volume concentration of 38% is reached - then it dropped drastically and the whole composite became an electrical conductor. Since Garland's work, many other researchers have reported different sharp transition from insulators to conductors at different volume concentrations (ref. 8-15).

Among their studies, Dearaujo and his co-investigator (ref. 10) had found that normally at least 40% volume fraction of metal fillers was needed in order to make a composite conductive. In the curing process of a resin matrix, they also suggested that a slow curing rate at lower temperatures would greatly enhance the conductivity of the composite.

Recently, because short fiber reinforced composites can offer design flexibility, weight reduction, energy savings and high-volume production for structural applications, they are widely used in automotive, recreation, business machinery, electrical appliance, and military applications. Metal fiber reinforced composites become highly desirable to meet the aforementioned requirements not only for load-bearing capability but electrical conductivity as well, which normally metal particle reinforcement cannot achieve. However, not much work has been done in this area. Experimental data were found only limited to individual cases. Davenport (ref. 16) mentioned in his study that the metal fiber length (L) to diameter (D) ratio (known as aspect ratio) in a composite must have 100 or more in order to induce electrical conductivity. He demonstrated the electrical conductivity should be a function of L/D. In addition, the fiber packing density is a significant factor which is closely associated with the ratio of L/D (ref. 17). Bigg and Stutz investigated a stainless steel fiber (8 microns in diameter, aspect ratio: 750) reinforced ABS system, and found that the composite had an electrical resistivity of 0.70 ohm-cm at the fiber volume concentration of 1% (ref. 18). They also claimed in their research that a highly conductive composite can be achieved with a low concentration of metal fibers by simply using high aspect ratio fibers. Their work seems very promising, yet needs to be proved. Most of the metal fiber reinforced composites emphasized the electrical properties rather than the mechanical properties.

Nickel has long been considered as a preferred metal because of its low electrical resistivity. In this study, Inconel 601 nickel based fiber with a diameter of 8 microns and an aspect ratio of 125 was heavily used to reinforce a commercially available thermoset polyester resin. Composite samples were made in coupon shapes depending on the test requirements. Both mechanical and electrical measurements were further conducted to help understand the micromechanical behavior as well as electrical conductivity.

SPECIMEN PREPARATION AND TESTING

Chopped Inconel 601 metal fibers were donated by Bekaert Fiber Technologies. To prevent the sizing effect from the interfacial bonding between fiber and resin matrix, a thin water-soluble PVA (polyvinyl alcohol) coating originally attached to fibers was removed from Inconel fibers prior to the process. Fiber volume concentration, varied from 0% to 50%, was carefully controlled as a material parameter to conduct this study. Metal fibers were completely mixed with an appropriate amount of polyester resin and MEKP (Methyl Ethyl Ketone Peroxide) curing agent in a chemical beaker based on a predetermined volume ratio. The mixture was then poured into an aluminum mold for cure. Traditional compression molding practice was employed in the curing process; pressure was around 17 psi (1.17×10^5 Pa) and temperature was set at 356°F (180°C). Specimen dimensions were carefully prepared according to ASTM standard test methods; they were 6 x 3/4 x 1/8 in. (152.4 x 19.05 x 3.18 mm) size for tensile test, 5 x 1/2 x 1/4 in. (127 x 12.7 x 6.35 mm) size for Izod impact test, and 4 x 1/8 x 1/2 in. (101.6 x 3.18 x 12.7 mm) for flexure bars. The tensile and flexure tests were performed in a screw-driven computer-assisted Satec testing machine. A testing speed of 0.1 in./min. (2.54 mm/min.) was used for tensile and flexure tests. The ASTM method D257 was also followed to measure the volume resistance of each sample. The test data were collected and discussed in the following sections.

RESULTS AND DISCUSSION

Tensile test data, as shown in Table 1, have demonstrated that fiber concentration can indeed increase the tensile strength of the composite. Young's modulus is also improved as well. It is interesting to note that the small fiber concentration at the ratio lower than 10 volume percent will not contribute to the increase of entire tensile strength. According to the study, fiber concentration at 25 % has the maximum UTS. It is found that fiber orientation along the pulling direction will have significant effect to tensile properties. Since the specimens are prepared through a casting process, the fiber orientation in all directions is assumed the equal. Impact test data (in Table 2) reveal that impact strength increases with the addition of metal fibers. However, there is a limitation set at 35%. Low fiber concentration impairs the impact strength of the composite. Optical microscopy indicates that because of the existing of metal fibers, small air bubbles are attached to fiber ends, which is believed to be responsible for the degraded impact strength. Three point (flexure) test data show that fiber fillers can improve the flexural strength and tangent modulus of the composite, as shown in Table 3. It is also noticed that metal fibers can dissipate some energy in a crack propagation.

In other words, with the addition of metal fibers the crack pattern of a given composite shifted from a pure tension failure mode toward a more shear failure mode, which increases the flexural properties. Fiber pull-outs and fiber breakage are some evidence. In the electrical measurements, a critical fiber concentration is recorded. Electrical resistivity of 1.0 ohm-cm is measured at the fiber volume ratio of 45%, which is unexpectedly high. Figure 1 shows fiber concentration below 30%; the electrical resistance remains almost constant, that is the composite is still an electrical insulator. In this study, metal fiber reinforced composites did undergo a sharp transition which is in concert with Garland's work (ref. 7).

CONCLUSION

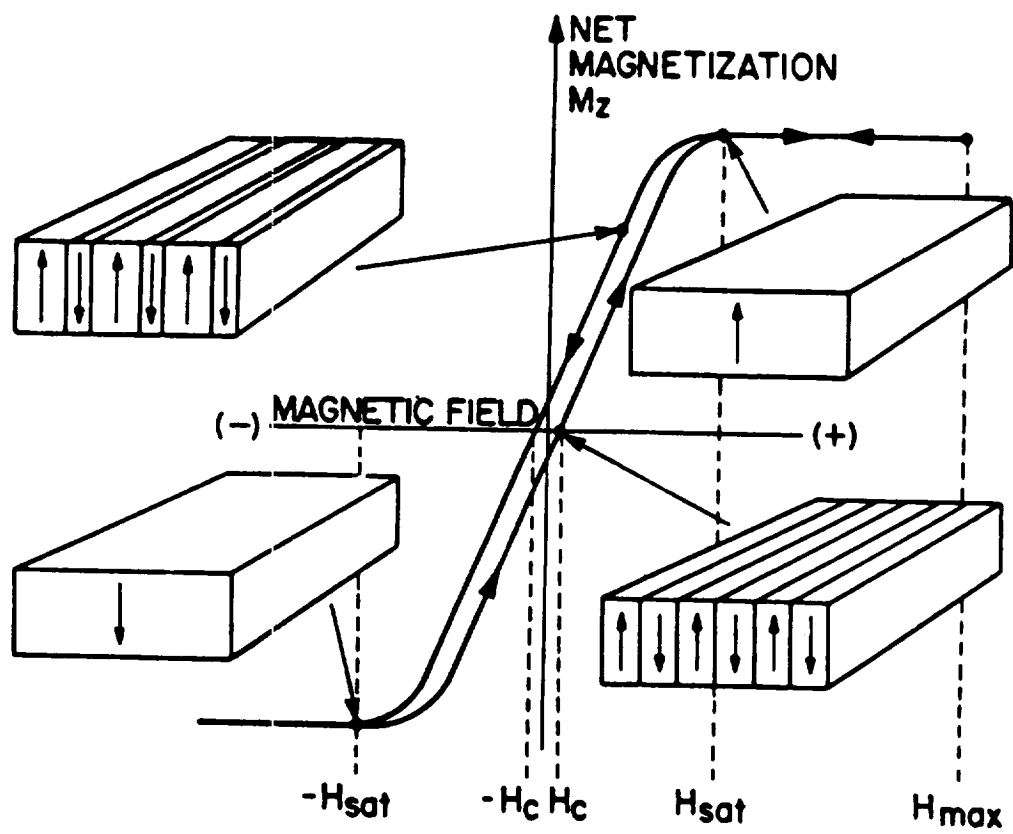
Because of excellent electrical conductors, metal fibers are suitable additives for inducing electrical conductivity in traditionally known insulators, polymer materials. Inconel metal fibers, although proved to be effective reinforcing elements, are considerably more dense than expected. It is found, during this study, that the explanation of mechanical properties is often difficult to make because it involves many unseen factors such as stress concentration, orientation effect, viscoelastic behavior etc. While significant progress has been made, much work still needs to be done. A systematic approach including experimental and theoretical techniques should be developed to help understand the micromechanisms and to clarify the interrelationships among structure, property and processing. Several factors such as fiber concentration, fiber aspect ratio, compatibility between fiber and matrix can then be studied under this guidance. The aforementioned suggestions, if applicable, may lead to a complete data bank setup which may eventually benefit all the designers, engineers, and scientists who are using conductive composites in their work.

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Experimental Determination of Material Damping Using Vibration Analyzer

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ABSTRACT

Structural damping is an important dynamic characteristic of engineering materials that helps to damp vibrations by reducing their amplitudes. In this investigation, an experimental method is illustrated to determine the damping characteristics of engineering materials using a dual channel FFT (Fast Fourier Transform) analyzer. A portable Compaq III computer which houses the analyzer, is used to collect the dynamic responses of three metal rods. Time - domain information is analyzed to obtain the logarithmic decrement of their damping. The damping coefficients are then compared to determine the variation of damping from material to material. The variations of damping from one point to another of the same material, due to a fixed point excitation, and the variable damping at a fixed point due to excitation at different points, are also demonstrated.

INTRODUCTION

A body once set to vibrate freely, will not do so indefinitely. The amplitude of the oscillation gradually decreases to zero as a result of friction. The body is said to be damped. An undamped material, once excited, will oscillate indefinitely with a constant amplitude. Any physical system, however, possesses some degree of damping forces which cause energy to dissipate during a cycle of vibration. The rate and the amount of this dissipated energy depend upon the physical and geometric properties of the material.

Damping of a vibrating material may be of two types: external and internal damping. External damping refers to separate energy absorber units which are added to the system for reducing resonant vibration. The internal damping which is an inherent material property causes heat build-up in a material due to the absorption of energy during a cycle of vibration. In this investigation only internal damping of materials is considered.

Damping forces can be used in an analytical model to determine structural dynamic response (Reference 1). A viscous type of damping which assumes that the forces are proportional to the magnitude of the velocity and opposite to the direction of motion is generally used in a mathematical model to consider its effect on the structural response. The inclusion of such an effect in a model, however, does not make significant variation in the quantitative magnitude of the dynamic properties than that of an undamped case. For instance,

the damped natural frequency only slightly differs from the undamped frequency of most real structures. This is partly because of the low range of damping co-efficients(less than 20%) present in a real structure.

The importance of damping characteristics, however, is more significant in studying or selecting new engineering materials. Current trends of producing sophisticated, high - performance material to replace traditional ones need more attention towards the dynamic performance specifications. An evaluation of the dynamic damping properties can be a critical factor in the material selection process. A material which damps off more quickly would obviously be a better choice than one which oscillates longer once it has started vibrating after the excitation force is removed. The large amplitude, at resonance, which decays faster can be identified as a good dynamic performance material.

As stated before a dual channel FFT (Fast Fourier Transform) analyzer is used in this investigation to determine the internal damping of different materials. The damping ratios of three rods, a copper, a steel, and an aluminium rod, are measured. The logarithmic decrement procedure of calculating damping is used as is found in literature (Reference 1), and is obtained from the time-domain information.

EXPERIMENTAL METHOD

Material and Test Set up:

This experiment consists of a testing rod, an input trigger source to excite the rod, an output device to measure the response signal, and an analyzer to collect and analyze the time - domain waveforms. A schematic diagram of the instrumental set-up is shown in Figure 1.

Three rods of identical length and diameter are selected in this experiment for studying and comparing the damping of each. The length and diameter are kept constant in order to see the effects of other material properties, such as mass and stiffness, upon the damping of the rods. The physical parameters of the rods are presented in Table I. SI unit conversion of each of the parameters in the table is listed inside a parenthesis. The moduli of elasticity of the rods are assumed to be equal to their respective typical values as obtained from the literature (Reference 2).

The rods are tested under identical support conditions as shown in Figure 1. Sponge foams are used to support the rods. This is done in order to avoid the bouncing effect which occurs due to use of rubber bands. Eleven points are marked off on each of the rods thus dividing them into 10 segments of approximately 2 inches each. Each of the points are numbered as shown. The numbering is required for identifying response or excitation location in the rods.

The trigger, a hammer, is connected to one channel of a dual channel analyzer and the other channel is connected to the output device, an accelerometer. The modally tuned hammer triggers the analyzer's mode of operation. This means that as soon as the hammer strikes the rod, imparting an impulsive signal, the analyzer starts collecting samples from the input and output sources. Here the impulsive signal refers to a single strike by the hammer instead of multiple strikes. Care is taken to make sure that this is the case because multiple strikes will generate multiple impulses which will interfere with the desired results. The free vibration generated by the rod after a single strike of the hammer is measured through the output device.

The signal generated by the impulse hammer and the response accelerometer are analogs in form and are digitized by the ADC (Analog to Digital Converter), then displayed on the computer screen. A typical time-domain signal output response of the aluminium rod is shown in Figure 2. This signal shows the fluctuating movement of the rod as it decays with time. This decay of the amplitude of motion describes the damping of the system.

For a linear system of vibration the ratio of the amplitude for any given cycle of vibration to the amplitude of another cycle is a constant. This constant is called the logarithmic decrement δ , which is defined as proportional to the natural logarithm of the ratio of two amplitudes which are apart at a multiple of time period as in Equation 1. Here Z_0 is the height of the peak of one of the periods of motion, and Z_n is the height of the peak after n cycles of vibration.

$$\delta = -\frac{1}{2\pi n} \ln\left(\frac{Z_n}{Z_0}\right) \dots \dots \dots (1)$$

The determination of damping, therefore, requires the measurement of two peak amplitudes, Z_0 and Z_n and the number of periods in between them, as shown in Figure 2a. The illustrated method is based upon certain assumptions and has some limitations in measuring the damping of a material. These assumptions and limitations are listed below.

Assumptions and Limitations:

- (1) The relationship used to determine the decay rate of a motion is adequate for a system having damping ratio less than 20% of the critical damping. Critical damping is that type of system damping which once excited will result in a non-oscillatory motion, the magnitude of which decays exponentially with time to zero,
- (2) Peak amplitude of a period is measured from an acceleration response instead of a displacement waveform. This can be done because it is assumed that the decay rate of the acceleration response is identical to that of displacement response, since the displacement amplitude is a constant multiple of the acceleration amplitude. Actually the double integration of the acceleration signal results in a displacement signal with identical pattern but different amplitudes,
- (3) Inspection of the periodic variation of time waveforms are done by naked eye, and
- (4) The sample rate of the analyzer was adjusted according to the nature of the resulting waveform from the output signal. A lower sample rate filters out the high frequency response content resulting in a signal pattern in which the periods are visually identifiable. Sample rates are considered in such a way that the definite pattern of periods are recognized, which may restrict the collections of some unrecognized frequency contributions.

RESULTS AND DISCUSSIONS

The time-domain responses of the rods are analyzed to compute the damping ratios of the testing materials. The damping ratios for the aluminium, steel, and copper rods are compared in Table II. The data are taken under identical support and excitation conditions. The accelerometer is kept fixed at the mid point of the rod, marked #6 on Figure 1. The

responses at this point are collected by successively exciting each of the eleven points on the rods. A typical signal for each of the materials is shown in Figures 2 to 4. To measure the accurate peak values and periods of the signal a portion of the waveform is expanded, as shown in Figure 2a.

The data obtained from this experiment are statistically analyzed to determine the differences among the groups. The analysis of variance (ANOVA) for the damping data of Table II shows that the damping ratios for each of the materials are significantly different. The probability that the mean values of these materials are equal is 0.003 (0.3%). As expected, the test result also shows that the mean value of the damping ratios for copper is greater than steel which has a mean value greater than that of aluminium. This means that if these rods were to be displaced equally under identical conditions and made to vibrate freely then the copper rod would stop vibrating fastest, the steel second fastest, and the aluminium rod third. Lower damping ratio means longer decay time. The deviations of the damping ratios from the respective means are also computed in Table II. This coefficient of variation is calculated as the standard deviation divided by the mean and multiplied by 100 (Reference 3). The high degree of dispersion of damping ratios from point to point for each case supports the conclusion that the decay rate varies from point to point of the rods.

Statistical analysis is performed to find out how much of the variation in the damping is due to differences between a fixed point trigger and a fixed point response. The damping ratios for these two cases are determined under the following conditions: (i) the triggering is at a fixed position (#6) while response is measured at different points, and (ii) the accelerometer collects the response at a fixed point (#6) while excitation is made at various positions. The data for this comparison are tabulated in Table III. Analysis of variance of this data is made by excluding the damping ratios of the points where the rod rests on the supports. This exclusion is done because of the greater variation of results at these two points, which may be due to the external influence of the sponge foam. It is found from the analysis that the damping ratios obtained from the two cases (i) and (ii) are not significantly different. The level of significance for this analysis is 0.2412 (24.12%). The coefficient of variation among the points of a single rod, however, is greater for fixed point triggering than for fixed point response. From the data it can be concluded that the response at one point due to excitation at another point is the same when the positions of the input and output devices are reversed. Moreover, it can also be concluded that the reciprocity between the response and trigger sources can be used to measure the damping of a linear system.

To understand the causes of variations of damping ratios among the different points of a single material, further analysis is performed. The sample rate which dictates the number of samples to be collected by the analyzer controls the frequency content of the output signal. For a lower sampling rate value, only the low frequency oscillation patterns can be measured while the contributions of the higher frequencies are truncated. Figure 5 shows a low frequency oscillation of the steel rod when the samples are collected at a rate of 250 per seconds. For a sample rate of 1200 per sec. the response collected for the same rod, as shown in Figure 3, differs significantly from the response with lower rate. This is because the higher frequency responses are also collected by the analyzer, as a result of which an unclear or fuzzy graphical reading is observed. In other words, in the second case, the total response includes the contributions of many modes of vibration. Modes are uncoupled dynamic parameters which describe the vibration of a physical structure (Reference 4). The variation of damping ratios when sample rates are changed is shown in Table IV. The clearer the response, the easier it is to identify periods. This in turn increases the accuracy of computing the damping ratio. The clarity of the response depends not only on the frequency content of the signal, but also, as shown in the Figures 2 and 3, on the material. For example, with a

low rate of sampling the steel rod gives a clear waveform pattern while, on the other hand, the aluminium rod shows a good waveform with a higher rate.

The modes of a structure are defined as the definite regular waveforms corresponding to each of its resonant frequencies. Modal analysis can isolate these resonant frequencies and the associated deflection pattern of Mode Shapes (Reference 4). The damping and frequencies for each of the modes of vibration of each of the rods are tabulated in Table V. The results of the modal analysis as presented in this table show that the damping ratios vary with the modes. Also it is seen that higher frequencies have lower damping ratio and therefore take longer to damp off. Again in Table IV it is found that for a sampling ratio of 1500 which includes higher frequencies determines smaller damping ratio than when sampling ratio equals 250. This variation can be used to explain why the damping among different points of a single material is not a constant. At a particular point certain modes will affect the response while others may have no affect at all. This latter case depends on the location of the nodes. For example, when the response is taken from the midspan, the second mode has no influence since at this point a node exists. A relationship can be found between the material damping and the weighted contributions of the modal damping in order to determine real life material damping.

CONCLUSIONS

The logarithmic decrement procedure measures the damping ratios in a reasonably accurate way. The results are consistent to the expected physical behavior of the materials. The damping ratios for copper, steel, and aluminium are in a decreasing order. The significant variations of damping ratios among the materials as found in this experiment may be due to the differences in their physical properties, while the variation among the points of an individual material is due to the type of signal output of the system which eventually depends upon the modal behavior of the system. Statistical confidence interval of the damping ratios can be determined from the experimental results which then can be estimated as a damping ratio of the material. A weighted average of the modal damping can be used to determine the damping of a system. The damping ratio is a variable entity which will depend upon material type, support conditions, and the frequency content of the output signal.

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TABLE I. - PHYSICAL CHARACTERISTICS OF RODS

Material Type	Length inches (cm)	Area of Cross-Section Sq. in, (cm ²)	Weight Lbs. (N)	Modulus of Elasticity lbs./ in ² (Mpa)
ALUMINIUM	25.47 (64.7)	0.20258 (1.307)	0.51609 (2.29)	10x10 ⁶ (0.6895 x 10 ⁵)
STEEL	25.35 (64.4)	0.1995 (1.2868)	1.416 (6.299)	30 x 10 ⁶ (2.0685 x 10 ⁵)
COPPER	25.43 (64.6)	0.2186 (1.4103)	1.753 (7.797)	17 x 10 ⁶ (1.172 x 10 ⁵)

TABLE II. - COMPARISON OF DAMPING RATIO FOR DIFFERENT MATERIALS: FIXED POINT RESPONSE DUE TO EXCITATION AT VARIABLE POSITIONS.

Position	Damping Ratio		
(Triggering point, Accelerometer point)	Aluminium Rod (Sampling Rate = 1500)	Steel Rod (Sampling Rate = 1200)	Copper Rod (Sampling Rate = 1500)
(1,6)	0.00400	0.00138	0.00900
(2,6)	0.00560	0.01140	0.00400
(3,6)	0.00354	0.00051	0.00268
(4,6)	0.00410	0.00820	0.01140
(5,6)	0.00306	0.01080	0.02200
(6,6)	0.00400	0.00052	0.01134
(7,6)	0.00491	0.01100	0.01625
(8,6)	0.00550	0.00910	0.01209
(9,6)	0.00250	0.00080	0.01409
(10,6)	0.00180	0.01060	0.00380
(11,6)	0.00390	0.00110	0.00868
Mean	0.003901	0.005946	0.0105
Coefficient of Variation (%)	28.65	79.42	52.72

TABLE III. - COMPARISON OF DAMPING RATIOS FOR ALUMINIUM ROD:
FIXED POINT TRIGGERING VS. FIXED POINT RESPONSE (Sampling Rate equals 1500)

Fixed Point Trigger		Fixed Point Response		
Position (Trgr, Accl)	Damping Ratio (a)	Position (Trgr, Accl)	Damping Ratio (b)	Ratio (a/b)
(6,1)	0.00400	(1,6)	0.00400	1.00
(6,2)**	0.01700	(2,6)	0.00560	3.03
(6,3)	0.00490	(3,6)	0.00354	1.38
(6,4)	0.00218	(4,6)	0.00410	0.53
(6,5)	0.00508	(5,6)	0.00306	1.66
(6,6)	0.00400	(6,6)	0.00400	1.00
(6,7)	0.00499	(7,6)	0.00491	1.01
(6,8)	0.00670	(8,6)	0.00550	1.22
(6,9)	0.00420	(9,6)	0.00250	1.68
(6,10)**	0.01398	(10,6)	0.00180	7.76
(6,11)	0.00496	(11,6)	0.00390	1.27
Mean	0.0065445		0.003901	
Coefficient of Variation(%)	67.06		28.65	28.65*

** Rods are supported at these points.

* Excluding support ratios.

TABLE IV. - COMPARISON OF DAMPING RATIO FOR STEEL AND ALUMINIUM ROD WITH DIFFERENT SAMPLING RATES (SR): FIXED POINT RESPONSE

Position (Trgr., Accl.)	Aluminium Rod		Steel Rod	
	Damping Ratio when SR = 250 (Samples/sec)	Damping Ratio when SR = 1500 (Samples/sec)	Damping Ratio when SR= 250 (Samples/sec)	Damping Ratio when SR = 1200 (Samples/sec)
(1,6)	0.00552	.004000	0.10280	0.00138
(2,6)	0.01194	0.00560	0.07600	0.01140
(3,6)	0.00231	0.00354	0.0930	0.00051
(4,6)	0.00766	0.00410	0.09600	0.00820
(5,6)	0.00088	0.00306	0.08100	0.01080
(6,6)	0.00192	0.00400	0.08030	0.00052
(7,6)	0.00597	0.00491	0.09300	0.01100
(8,6)	0.00640	0.00550	0.09030	0.00910
(9,6)	0.00825	0.00250	0.09500	0.00080
(10,6)	0.02378	0.00180	0.07900	0.01060
(11,6)	0.00416	0.00390	0.09800	0.00110
Mean	0.00716	0.00390	0.08949	0.00595
Coefficient of Variation(%)	84.65	28.65	9.5	79.42

TABLE V. - COMPARISON OF MODAL DAMPING RATIOS AND FREQUENCIES FOR DIFFERENT MATERIALS

Mode of Vibration	Types of Rods					
	Aluminium		Steel		Copper	
	Frequency (Hz)	Damping Ratio	Frequency (Hz)	Damping Ratio	Frequency (Hz)	Damping Ratio
1	135.15	0.003990	115.57	.224100	106.39	0.000869
2	371.88	0.000466	385.52	0.002636	292.50	0.000472
3	729.25	0.000093	751.49	0.000207	571.89	0.0001327
4	1201.00	0.000309	1234.00	0.000055	1399.00	0.0000794
5	1780.00	0.000477	1842.00	0.000050	1939.00	0.0003735
Mean		0.001067				0.00039
Coefficient of Variation		137.0%		---		73.3%

--- Not Computed

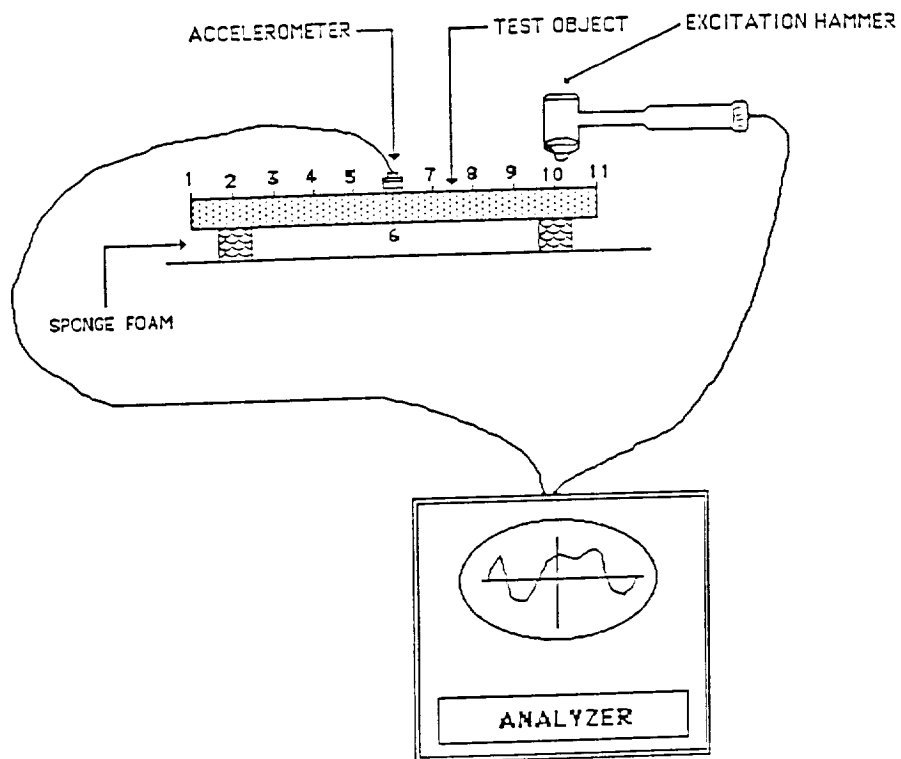


Figure 1: Schematic Diagram of the Test Set-up.

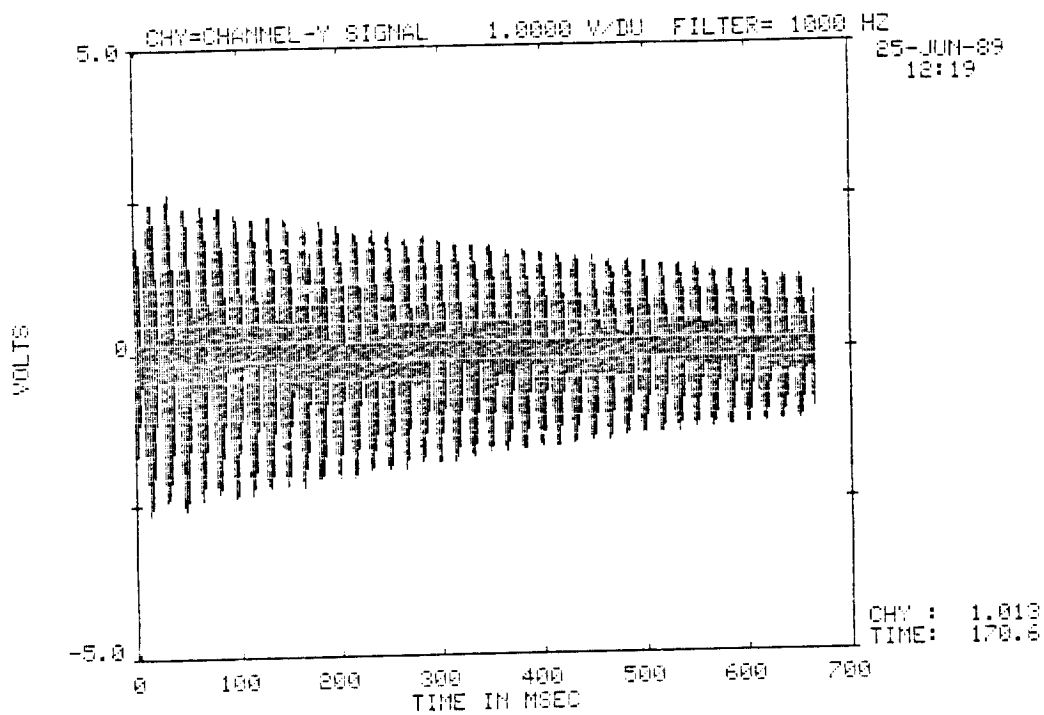


Figure 2: Free Vibration Response for Aluminium Rod (Accelerometer at Point #6 and Trigger at Point #3).

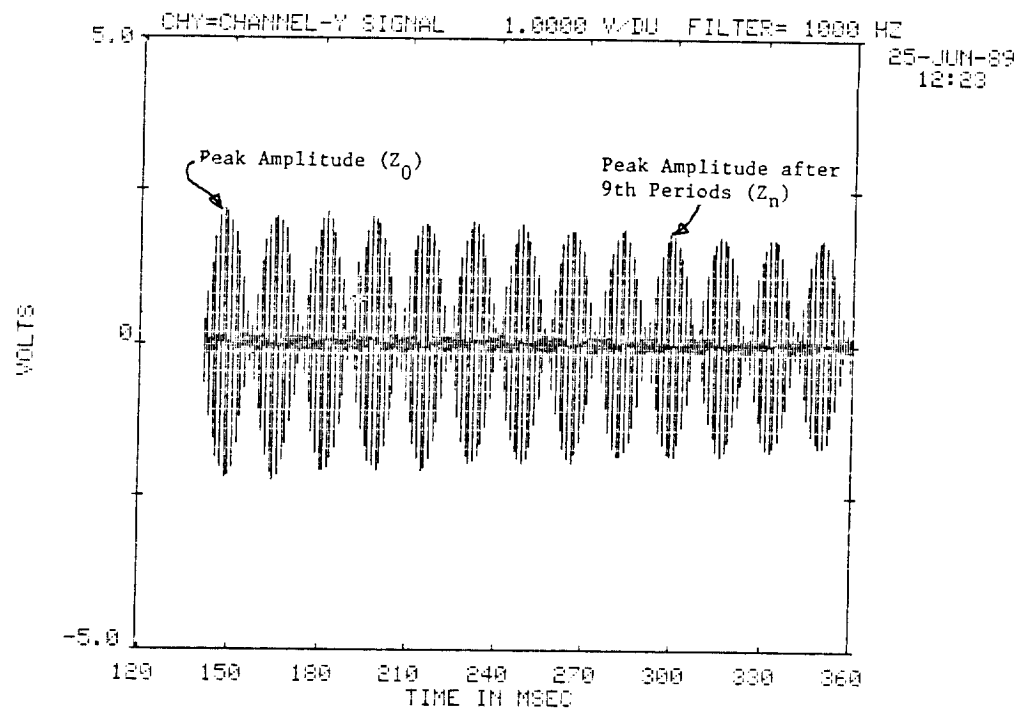


Figure 2a: Expansion of Figure 2.

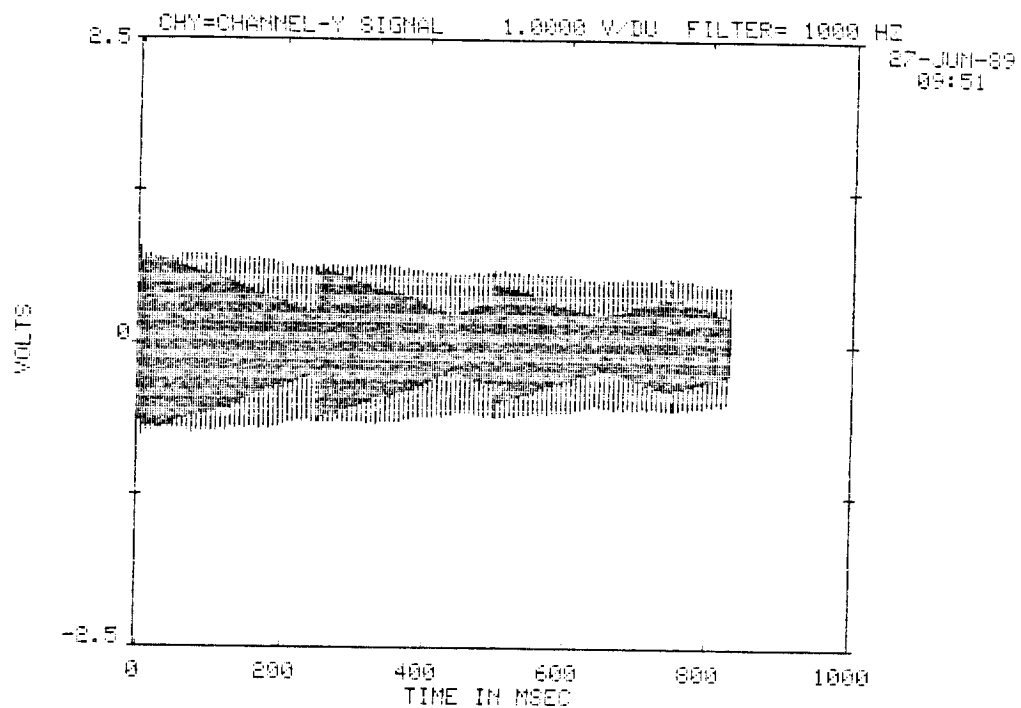


Figure 3: Free Vibration Response for Steel Rod (Accelerometer at Point #6 and Trigger at Point #3).

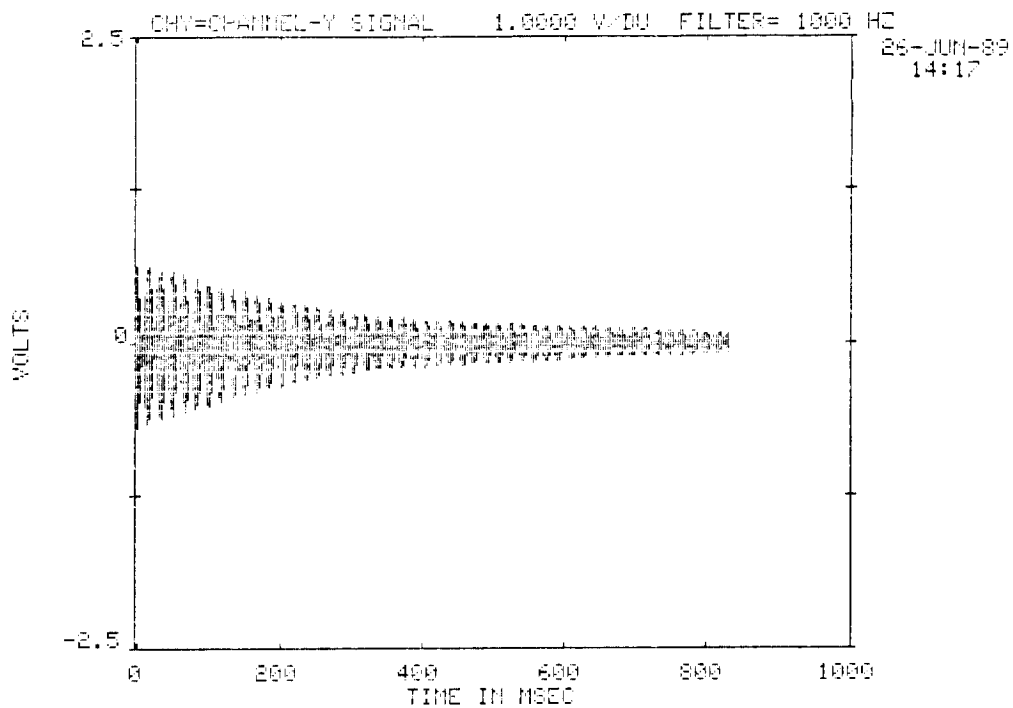


Figure 4: Free Vibration Response for Copper Rod (Accelerometer at Point #6 and Trigger at Point #3).

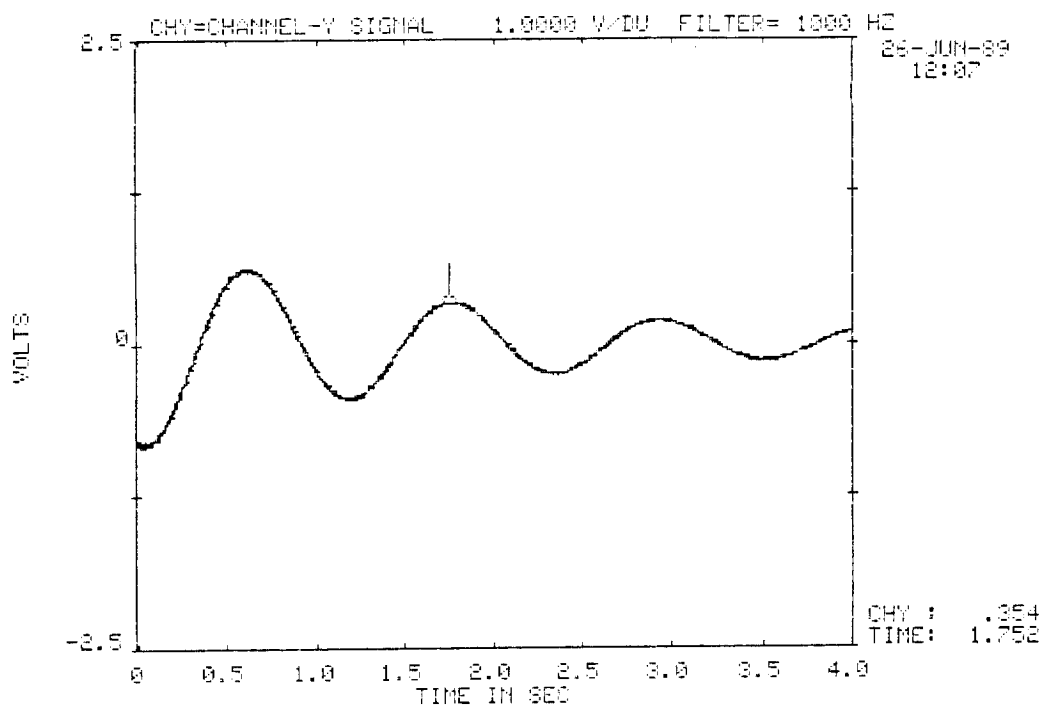


Figure 5: Damped Vibration of Steel Rod when Sample rate equals 250 (Accelerometer at Point #6 and Trigger at Point #3).

RECYCLING WASTE-PAPER

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ABSTRACT

Perhaps 80% of papermaking energy is expended in chemical pulping of vegetable cellulose, a natural polymer. Commercial supplies of wood, bagasse, cotton and flax are valued as "renewable resources" and "bio-mass" assets; however, few enterprises will salvage waste-paper and cardboard from their "trash." A basic experiment in the Materials Lab uses simple equipment to make crude handsheets. Students learn to classify "secondary fibers," identify "contraries," and estimate earnings.¹

INTRODUCTION

The U.S. still has 70% of those forests existing at Columbus'time, and about 50% of New York State is forest. Such vegetation is a prime national resource, a renewable raw-material for lumber, pulp, paper, plastics, and fuel. These industries represent major job-opportunities for mechanical technologists in production, process and maintenance. Yet, materials labs tend to neglect non-metals, especially common cellulose.

Pulp and paper production requires vast amounts of air, energy and water. Surging demands threaten to outstrip our plantations or degrade our environment, despite ingenious genetics and fiber conservation cited by ecologists.² Paper consumption, now estimated at 100-million annual tonnes, may reduce today's forests from 9.7 square meters per person to 7.7 (80%) by the year 2000.

Solid wastes are about 50% paper, increasing three times faster than U.S. population. Overcrowded dumps and smoky incinerators are now unacceptable. Most recycling involves only "pre-consumer" waste (mill-broke, rejects, trim) and comprises only 25% of "consumer-products" paper. The challenge is to salvage "post-consumer" waste.³ The public should be educated and motivated to segregate marketable grades and to accept subsequent products.⁴ Meantime, technologists can be trained to promote recycling by utilizing available and inexpensive equipment.

TERMS

A different pulp and paper vocabulary emerges and new properties are introduced. Waste paper is "secondary-fibre". Color is specified by hue-depth-brightness, with metamerism from light-heat-humidity. Fast means permanent. Wires and felts are machine-clothing, and white-water is recycled machine-drainage. Fines are fiber particles, and broke is sheet-scrap. Converting is subsequent processing. Glassine is film from hard calendering. Fiber is floc, staple, fibrid, fibril, and tow. Non-wovens are formed on cylinders and fourdriniers.

Couching and picking affect sheet morphology. Deinking and bleaching affect secondary-fiber's softness and tenacity. MIT-fold (fatigue endurance), Elmendorf (impact tear propagation), Mullen (burst strength), Patterson (puncture), Taber (sheet stiffness), Finch (edge-tear), and Canadian Std. Freeness (pulp drainage) are properties. Measurements include caliper (thickness), basis weight (density as U.S. lb/ream), denier (grams/9000 m of yarn), sheet appearance (holes, feel, smoothness, drape), and absorbance (wicking).⁵

PAPER GRADES

Pulps are classified by manufacturing process, timber source, or color:

1. Sulfite - made with sodium or calcium bisulfite; needs clean-up of plant waters; expensive and soft.
2. Sulfate (Kraft) - made with sodium sulfide and hydroxide; needs air cleanup; strong and cheap.
3. Soda - made with sodium hydroxide; cheaper than sulfite.
4. Semi Chemical - partial digestion and steam expansion.
5. Mechanical Groundwood - sawdust or wood flour; weakest, cheapest, harshest.^{6,7}

Timbers are hardwood (deciduous; harsher, cheaper) or softwood (coniferous, evergreen; softer, costly) from the North (cold, slow growth; thin, supple fibers) or from the South (fast growth; thick, harsh fibers).

Colors are white (fully bleached), tan (semi-bleached) or brown (unbleached). Bleaching enhances softness, hurts strength.

Prices of virgin pulp range from \$300-\$800 per short ton (+1.1023 for metric ton), compared to recycled fibers of \$30-\$300. A typical mill using \$2,000,000 of pulp/month could save \$100,000/month with 25% recycled "secondary fiber" priced only 20% less. Some cardboard plants run 100% recycled boxes, bought from local stores and scrap dealers. This is big business.⁸

PROCEDURE

In the mills, baled pulp and paper are hydrated and defibered to make "handsheets" for quality control tests, according to TAPPI Std. T-200. The conventional agitators (Valley® Beater; British Std.® Disintegrator) often take several hours. However, a Waring Blendor® takes 30-60 seconds, without presoaking! Blending involves intense fluid-shear in a 4-lobed canister, as 4-vortexes impinge. Thus, fiber clumps are unrolled rather than chopped to "fines" by impeller-blades.

Commercial labs make handsheets with vacuum-boxes (Valley® sheet mold) and steam-radiators (Williams® sheet-dryer).⁹ Waste-paper dealers use conventional lab-filtration setups (Ehrlemeyer flask, with Büchner funnel and filter-paper) requiring a vacuum pump or eductor nozzle (run tapwater to sewer).¹⁰ However, the pocket-sized "scoop and press" (Ciba-Geigy® color sampler) can make 8 cm dia. sheets, quickly and easily. Thinner sheets are made by diluting the pulp-slurry

consistency. In a pinch, use a flat-bottomed strainer or evaporate a jar of slurry to leave a fiber-sheet.

A typical recipe is a) Tear a tabcard (83 x 187 x 0.02 mm thick) into postage-stamp pieces; b) Drop into Blendor®, with 50 ml. water (23°C); c) Blend at low-speed for 30 sec; d) Pour slurry into shallow bowl; e) Look for complete defibering of "pulp-bundles"; f) Scoop and press to de-water the "pulp-patty" and lift the new sheet, which sticks to the press rather than the screen-knuckles; g) Dry the handsheet on hot-plate or electric-griddle; h) Use a lid to keep the sheet flat and aid inspection; i) View thick sheets by reflected light, to find "contaminants" (flakes of clay, specks of foil or film); j) Thin sheets are viewed by transmitted light, to find buried "contraries" (wet-strength clumps of fibers); k) Tiny balls of pernicious latex or tar are probed with a needle, pulling a fine filament like a spider web; l) As required, dyes and wet-strength can be tested for "bleachability;" m) Pulping time may be reduced, using hotter water.

CONCLUSION

Students should be instructed to write formal proposals to management, detailing each step of a careful investigation:

a) consistent monthly tonnage of available waste-paper, by grade; b) location and cost of baler; c) selection and cost of fiber-collectors and equipment-operators; d) selection of a reliable dealer and determination of minimum fiber price; e) method of shipment, ensuring plant security; f) estimated earnings and equipment payoff, including savings of trash-service. Busy bosses ignore casual suggestions.

RECYCLE SAMPLES 11,12

1. Newspapers - plain, printed, colored.
2. Magazines - pages only; no covers or staples; no coatings, clay or latex.
3. Envelopes and Stationery - white, colored, unbleached; no glassine windows or latex labels.
4. Creped wadding - facial, bathroom, towel, napkin tissues; white, pastel, deep-dyed; plain and wet-strength.
5. Computer scrap - printouts or tabcards; no carbons; white, pastel.
6. Kraft sulfate - bags, wrappers; unbleached, semi-bleached.
7. Boxboard - linerboard, corrugate; unbleached, semi-bleached; grey, brown.

EQUIPMENT AND SUPPLIES

1. Waring Blendor® - 120 v, 60 Hz, stainless steel canister.
 - a. Commercial Model CB-4 (TMI#52-4-2), 1 gal capacity (3.79 litres), 14,000-19,000 rpm, approved by Canadian Standards Association...\$700.

- b. Model 31-BL-92, 1 qt capacity (.95 litre)
2-speed.....\$100.
2. Sunbeam Griddle® - 120 v, 60 Hz
 - a. "M'sieur Crepe," cat.#30-10, 750 W, with thermostat control
and skillet lid.....\$ 20.
 - b. Sandwich grill or frypan.....\$ 20 - 40.
 - c. Laboratory hot-plate.....\$ 70.
3. Ciba-Geigy® Scoop and Press - stainless steel pans, nested.
 - a. Scoop approx. 8 cm I.D. x 2.2 cm depth, with wire screen
on bottom (80-mesh).
 - b. Press approx. 7.5 cm O.D. x 2.5 cm depth, with solid bottom
(deep-drawn).
4. Weigh Scale.
 - a. Ohaus Dial-a-Gram® balance, 310-g
capacity.....\$100.
 - b. Standard postal scale (oz, g).....\$ 10.
 - c. Regular dietitian's scale (oz, g).....\$ 20.
5. Coffee pot, with thermostat control.....\$ 20.
6. Containers (glass or plastic).
 - a. Graduated cylinder or flask (100 - 1,000 ml).
 - b. Battery jars or cut jugs (2-4 litre size).
7. Bleaches.
 - a. Hydrogen peroxide (3.0% H₂O₂).
 - b. Sodium hypochlorite (5.25% NaOCl).
8. Dissecting needle (biology probe).
9. Spatula (pallet-knife).

ABBREVIATIONS

- | | | |
|------|---|---|
| APPA | - | American Paper and Pulp Association, New York, NY. |
| API | - | American Paper Institute, Inc., 260 Madison Ave., New York, NY 10016. |
| EE | - | Environmental Educators, Inc., 1621 Connecticut Ave., NW, Washington, DC 20009. |
| EPA | - | Environmental Protection Agency, Washington, DC. |
| NARI | - | National Association of Recycling Industries, Paper Stock Div., 330 Madison Ave., New York, NY 10017. |
| NCRR | - | National Center for Resource Recovery, 1211 Connecticut Ave, NW, Washington, DC 20036. |

- TAPPI - Technical Association of the Pulp and Paper Industry,
1 Dunwoody Park, Atlanta, GA 30338.
- TMI - Testing Machines, Inc., 400 Bayview Ave., Amityville, LI.,
NY 11701.
- Prefixes - Centi (c), milli (m), page (p).
- Ream - Specific number of standard-size sheets; standard area
varies with paper type.
- Units - Gallon (4 quarts, 3.785 litres); gram (g); litre (l);
metre (m); mil (0.001 inch); ounce (oz) Avoirdupois
(28.35 g).

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PIEZOELECTRIC AND PYROELECTRIC EFFECTS
OF A CRYSTALLINE POLYMER

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SUMMARY

Polyvinylidene flouride (PVDF) is a crystalline polymer of both piezoelectric and pyroelectric nature. Piezoelectricity produces electrical signals when mechanically deformed, and pyroelectricity is the electrical polarization induced by thermal absorption in crystals. To demonstrate the piezoelectric effect PVDF is subjected to impact loads which produce electrical charges proportional to mechanical stresses. A heat source was used to demonstrate the pyroelectric nature of PVDF. The rise in temperature due to absorbed energy by the polymer produces electrical output. The qualitative test results obtained are graphically reproduced.

INTRODUCTION

Piezoelectricity is a material characteristic of certain crystalline polymers that produce electrical signals when mechanically deformed and experience dimensional changes when subjected to an electrical field. Pyroelectricity is the electrical polarization induced by thermal absorption in crystals.

This experiment deals with the demonstration of the above phenomena using Kynar Piezo Film, a specially treated polyvinylidene fluoride (PVDF) film manufactured by the Pennwalt Corporation. The special treatment consists of aligning the polar axes of individual crystallites and coating both sides of the film with aluminum or chromium for electrical contact.

To demonstrate the piezoelectric effect, the PVDF film is subjected to impact loads which produce electrical charges proportional to the mechanical stresses. A heat source is used to demonstrate the pyroelectric nature of PVDF. The absorption of thermal energy by the polymer produces an electrical output; this effect reverses when cooling.

Some of the most common applications of piezoelectric films are found in acoustics, ultrasonics, optics, transducers, and medical instrumentation.

IMPACT TESTS

Test Specimens

The specimens used in the tests were 1.0 inch square pieces of treated PVDF film. Copper electrodes 1/8 in. x 3 in. were attached to either side with an electrically conductive adhesive. The specimens were then placed in between two pieces of nonconducting, transparent protective plastic film.

Apparatus

PVDF specimen, 3/8 in. steel ball, power supply, chart recorder, and weights.

Test Procedure

The steel ball was securely attached to the center of the square film. The electrodes attached to the specimen were connected to the chart recorder. The chart recorder was set at a speed of 25 divisions per second and a sensitivity of 50 millivolts per division.

Various weights were dropped from a certain height onto the ball attached to the film. The impacts transmitted through the ball created point loads on the specimen which in turn produced electrical signals on the chart recorder. Signals were recorded five times for each weight.

The obtained electrical output is a representation of the impact load experienced by the film. The impact can be measured qualitatively from the amplitude of the signals.

Interpretation of a Sample Output

As shown in figure 1, the impact will cause both positive and negative amplitudes. For this reason, only the first crest (arbitrarily considered positive) was used to indicate the output from the film.

See table 1 for experimental data from impact tests. Only a portion of the data is given.

THERMAL TESTS

Test Specimens

The specimens used in these tests were identical to those used in the impact tests.

Apparatus

PVDF specimen, thermometer, heat source, ice, power supply, chart recorder, and a stopwatch.

Test Procedure

The electrodes extending from the specimen were connected to the chart recorder. The chart recorder was set to a speed of 25 divisions per second and a sensitivity of 20 millivolts per division.

The film was subjected to a cycle of thermal stresses as follows: The specimen was heated for 30 seconds while recording its temperature every 10 seconds. Then ice was placed on the film for the next 30 seconds while the film's temperature was recorded every 10 seconds. Heat was again applied for 30 seconds while taking temperature readings. After 30 seconds, the heat source was removed and the specimen was allowed to cool for 30 seconds while taking the temperature readings every 10 seconds. This procedure was repeated three times.

Interpretation of a Sample Output

As shown in figure 2, thermal stress on the specimen can result in a constantly fluctuating electrical signal. In such cases, the mean amplitude should be considered for a qualitative evaluation.

See figure 3 for a graphical representation of the electrical output of the PVDF film. See table 2 for quantitative test data.

RESULTS AND DISCUSSION

Impact Tests

The electrical output from the impact tests is a reflection of the structural changes experienced by the polymer. According to figure 4, the electrical output is proportional to the impact of weights above 250 g. Due to insufficient stress, the linear relationship is not observed at lower weight ranges.

Thermal Tests

The electrical output from the thermal tests shows changes in heat flow to the polymer. According to figure 3 and table 3, the electrical output is extreme when changing from ice to the heat source or vice versa rather than continuous application of either although the temperature change was greater during continuous application of thermal stress in one direction.

TABLE 1 - IMPACT TESTS

Weight (g)	Trial	Output (mv)
90	1	200
	2	200
	3	200
245	1	250
	2	250
	3	250
251	1	250
	2	225
	3	250
268	1	250
	2	250
	3	250
354	1	550
	2	450
	3	650
359	1	550
	2	400
	3	400

All weights were dropped from
5.0 inches above the film.

TABLE 2 - THERMAL TESTS

	Time (min:sec)	Temperature (°C)	Electrical Output (mv)	
			Mean	Maximum
Heat Source	0:00	21.3	40	200
	0:10	24.3	30	100
	0:20	28.3	0	90
Ice	0:30	31.6	-300	-500
	0:40	31.0	-10	-80
	0:50	27.5	-5	-45
Heat Source	1:00	24.2	40	200
	1:10	24.6	10	160
	1:20	29.0	0	80
None	1:30	33.3	35	200
	1:40	32.5	20	100
	1:50	31.5	10	60

IMPACT TESTS

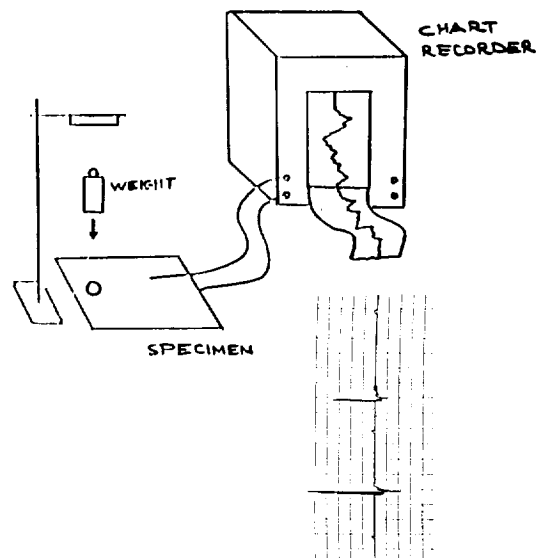


FIGURE 1

THERMAL TESTS

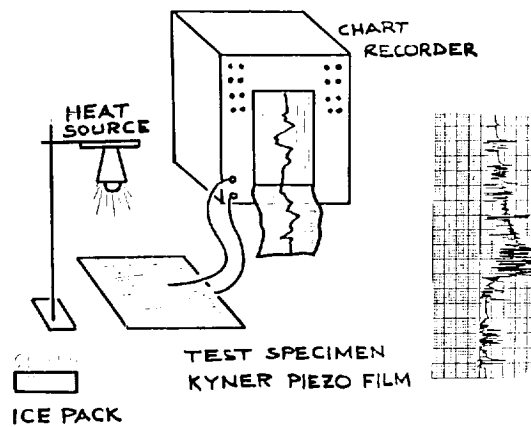


FIGURE 2

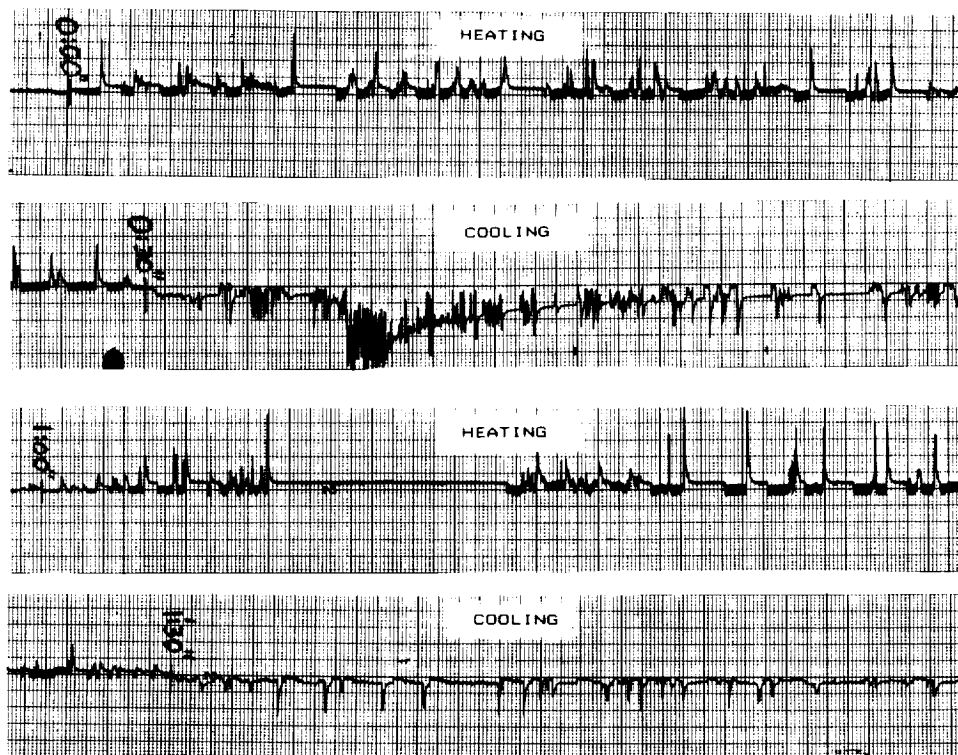


FIGURE 3

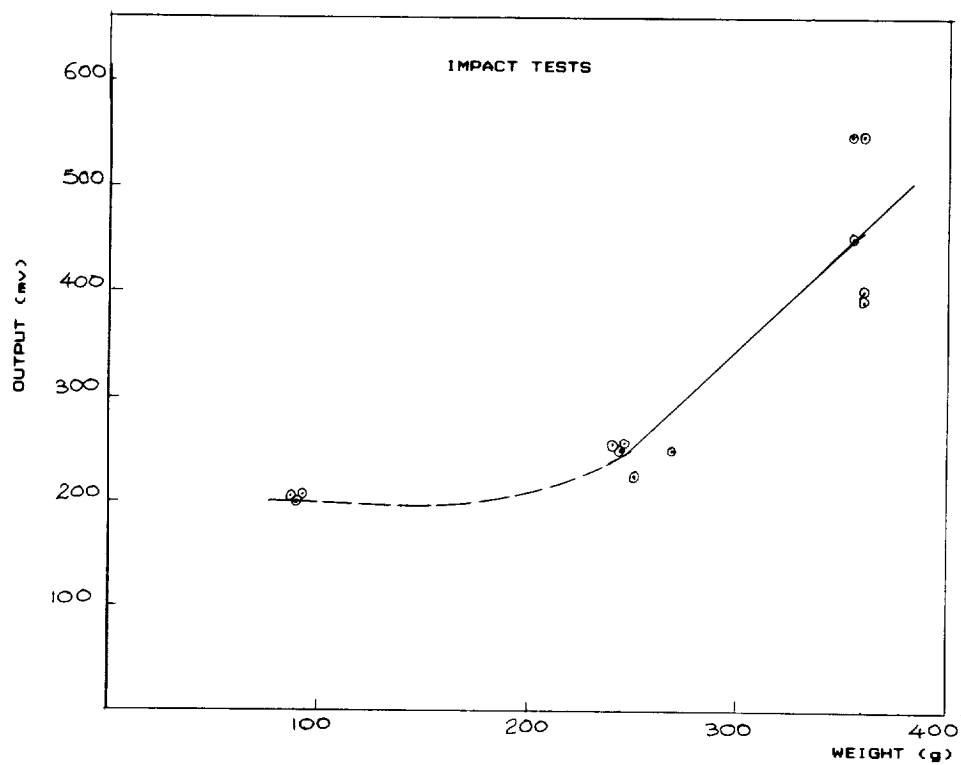


FIGURE 4

USING TEMPLATE/HOTWIRE CUTTING TO DEMONSTRATE

MOLDLESS COMPOSITE FABRICATION

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PREREQUISITE KNOWLEDGE REQUIRED

A person duplicating the following experiment should have some basic skills with handtools and simple woodworking machines.

OBJECTIVE

The objective of this experiment is to provide a simple, inexpensive composite fabrication technique which can be easily performed with a minimum of equipment and facilities. This process eliminates expensive female molds and uses only male molds which are easily formed from foam blocks. Once the mold is shaped, it is covered with fiberglass and becomes a structural component of the product.

EQUIPMENT AND SUPPLIES

Hand Tools

Flexible steel tape
Combination square w/centering head
Twist drills, 9/64", 1/4"*, and 3/8"
Scratch awl
Handscrew clamps w/10" openings
Level, 24"

Wood rasp
Wrench, 1/2"
Adjustable drafting curve
Scissors
Metal straight edge, 36"
*Spade bit, 11/16"

Wood Products

1 - 4' x 8' sheet, 1/8" hardboard
*1 - 2" x 4" x 24"
2 - 14" x 15", 3/8" plywood
6 - 3/4" x 2" x 2" blocks

1 - 10" dowel rod, 3/8"
1 - 18" dowel rod, 1/4"
with one end sharpened
2 - boxes toothpicks

Machines

Scroll saw
Drill press
Belt sander

Large portable fan
Vacuum cleaner

Metal Products

- 1 - 12" all-thread 3/8" with 2 nuts and washers
- 12 - 2 1/2" double headed nails
- * 2 - 20" - 11/16" O D electrical conduit
- * 2 - 2 1/2" machine screws with 2 wing nuts & washers
- *24" stainless steel aircraft safety wire, .041D

Miscellaneous

- | | |
|------------------------------|---------------------------------|
| 1 roll duct tape | 35" chalk line cord |
| 1 bottle rubber cement | 1 felt tip marker |
| 1 pint white latex paint | 1 paint brush, 2" nylon |
| *10' heavy gauge lamp cord | *1 variable voltage transformer |
| Dust mask, disposable | Rubber gloves, disposable |
| 4 plastic mixing cups, 8 oz. | Plastic coffee can lids |
| 10 stir sticks | |

Epoxy and Foam

- 1 - Safe-T-Poxy kit, 1 1/2 gal. (See section on Instructor Notes
- 1 - Epoxy pump or balance beam for info on using Safe-T-Poxy)
- 10 - sheets polystyrene insulation board, 2" x 2' x 8'
- 1 - sheet polystyrene insulation board, 3/8" x 2' x 8'

**Items marked with * are necessary for making and using the hot wire cutter. See section on Instructor Notes for details on the hot wire cutter.

PROCEDURE

Developing the Templates

Many foam plugs for composite products can be easily formed by hotwiring around templates placed on foam blocks. This moldless process works extremely well for prototypes, proof-of-concept products, and home/school one-of-a-kind projects. Two of the most popular items which can be hotwired are airfoils and boat hulls. The cutting and forming of a fourteen foot canoe hull was selected for the following experiment because of its simplicity. It is only necessary to

reproduce 1/4 of the canoe's contour because a mirror image can be made to achieve the total width of the templates. After 1/2 of the canoe hull is cut, another set of foam blocks is then cut by using the templates in the reverse order.

When selecting a canoe design, keep in mind that a white-water/river canoe has a rounded bottom and a lake/fishing canoe has a flat bottom. The cross-sectional contour may be taken from a production canoe by gauging the outside with a flexible, irregular curve and transferring it to paper. A CADD program can also be utilized by establishing the X,Y, and Z coordinates and allowing a plotter to draw the templates. In either case, the cross-sectional readings should be taken every ten inches starting at the center of the canoe and working outward.

Once the outside contour of each template is established, the inside contour is made by drawing a curved line two inches inside the outside curve. This two inch thickness is necessary to give the polystyrene arches enough strength to withstand the stress of laying fiberglass on their outside. It also allows enough thickness in some templates for double bolt holes at the centerline (see Photo 1 at the end of the experiment).

After the paper templates are drawn, they should be scissor-cut leaving about 1/4" border around the drawn outline. The template should then be glued to 1/8" hardboard using rubber cement. (Elmer's glue will cause the paper to wrinkle.) Cut out the templates using a scroll saw and then lightly sand the cut edge with a belt sander. It is critical that the edge be smooth to prevent the hot wire from hanging.

Identify each template with a number starting with #1 for the large center template and ending with #8 for the small end template. Mark a left and right symbol on each leg when the paper side of all templates is turned the same direction. Paint the opposite side of the templates with white latex paint so that the various marks can be easily read. Transfer the center line from the paper side to the painted side and then divide each leg into eight parts using the centering head on a combination square. Identify the lines that divide the parts with letters ranging from A to O. Line H will be the center line through which a 3/8" bolt hole will be drilled later. Drill 9/64" nail holes at lines A, D, L and O. Make sure each line on the painted side is identified the same on the opposite paper side and not on the opposite leg. The purpose of these lettered lines is to aid in controlling the speed when cutting the foam blocks.

The location of the bolt holes in the template is critical for the correct alignment of the foam. To position these bolt holes, each template should be temporarily attached (duct taped) to the jig table at its correct location (see Photo 2). A 10" long x 3/8" straight dowel rod with a level attached should be positioned between two templates and the ends moved up or down until the rod is level. (Two

people are required for this step.) The center line of the dowel rod should be marked across the template line H and then the procedure duplicated until all 3/8" hole centerlines are marked. Notice the double holes in the templates 3, 5 and 7.

Using the Templates

The positioning of the templates on the foam and keeping them turned the right direction is very important to keep the contour smooth. Begin with the #1 center template and the #2 for the first section to be cut (see Photo 3). Clamp five 2" x 2' x 8' foam blocks together using hand-screw clamps. Measure down from the top edge of the foam block and over from the end to locate the center point of the bolt hole. The position of this hole should allow the large template to come within 1/4" of the end of the foam block and 1/4" down from the top. Start the hole by piercing each side of the foam with a scratch awl; then use a sharpened 1/4" dowel rod to complete the hole by working from one side to the other. Push the 3/8" all-thread rod through the foam and allow it to stick out one inch on each side. The bolt hole in the foam will be much larger than necessary but it can be corrected later.

Attach the templates by placing them on the all-thread and securing them to the foam by hand tightening the nuts. (Use a washer between the nuts and the templates.) The all-thread must run horizontally and should be checked at this point by again measuring down from the top and over from the end. There is usually enough slack in the foam hole to allow some corrections to be made. When the all-thread position is correct, tighten the nut with a wrench until the foam just starts to compress. Check to make sure the templates are not reversed.

The final alignment is made by placing a level on the bottom ends of the templates and rotating them slightly on the all-thread until they are level. Force double headed nails through the template nail holes and into the foam. The foam blocks are now ready to hot wire.

Place the large foam blocks on a workbench and secure them by using large hand-screw clamps. Before starting the cut, make sure the throat of the cutter is deep enough to be able to go completely around the templates without having to back up. This may require rough cutting a corner or two from the foam block. It is best to have two people operate the cutter so that each can watch the cutting operation and keep the wire against his template. The person cutting around the largest template should call out the lettered lines as he reaches them. This allows the other person to gauge his cutting speed.

The outside contours are cut first and then the inside. The four corners should be square and the ends flat. Corners can be cut square by stopping the wire at the corner of the template, counting to three, which allows the center wire lag to catch up, and then proceeding

around the corner.

When the cuts are completed, it is impossible to remove the cross-section because it tapers both directions. It will be necessary to remove some of the uncut foam before the cross-section will slide out. Be careful not to ruin the large foam block for cutting some of the other cross-sections. When the cross-section is removed, it will look like the one in Photo 4.

The remaining cross-sections are cut using the same steps as above. When proceeding to the next set of templates (numbers two and three), it is best to set them in place on the jig table and then place them on the foam exactly in that position. Remember that the templates must always be turned the same direction and that they are reversed when cutting the opposite end of the canoe. When all cross-sections are cut, no two will be the same.

The polystyrene insulation boards are just under two inches thick and when five are put together the total thickness is 9-5/8". An additional foam piece must be cut from 3/8" insulation board by turning the cutter vertically in a vise and cutting around a single template (see Photo 5). The template is held in place by several pieces of duct tape looped with the adhesive side out and compressed between the template and the foam.

Gluing and Shaping the Cross-Sections

The body of the canoe can now be formed by resting the cross-section ends on the jig table and gluing the 2" pieces together. Since there are ninety foam cross-sections, the gluing will go much faster if two or more people participate. Glue the sections in sets of five plus the 3/8" spacer and hold them together with toothpicks. The glue should be Safe-T-Poxy as described in the Instructor Notes section and should be placed no more than 3/4" inside the outside curve of the foam pieces. These pieces are 2" thick and must be thinned to 1" during a later step. This is done with a small hot wire cutter which will not cut through the glue if it is allowed within an inch of the inside curve. When all sets of five are glued, then glue these sets together.

The end caps for gluing should be rough cut a little larger than the #8 template. Use 3/8" or thicker plywood with a 1/8" cord hole drilled in the center. Several 3/4 x 2" x 2" spacer blocks should be cut and a saw kerf cut to their centers to allow them to slip over the cord.

When all the foam pieces are glued and are in position, pass a heavy cord through the canoe body and through the center holes in the end caps. Secure each end of the string by tying it around a nail and then tighten the string by putting spacer blocks between the nail and

the end caps (see Photo 6). This unites the foam pieces into a solid unit which can be adjusted on the jig table.

Check the contour of the canoe bottom to make sure it is smooth with no low places. It may be necessary to put spacers between the foam blocks and the jig table to get the contour smooth. This can be done by releasing the string tension, removing some toothpicks, and raising or lowering the foam blocks. After the adjustments are made, the string is tightened again. Make sure that all foam pieces extend at least 1/2" beyond the edge of the jig table on both sides. This allows the fiberglass to drape straight down off the foam and not create a corner at the jig table. It is best if the canoe body is glued to the jig table to keep it from moving while forming and shaping.

The canoe ends are formed from solid scraps which were cut earlier from the foam cross-sectional arches (see Photo 7). The solid ends are easy to shape and provide additional flotation. Use Safe-T-Poxy and put it close to the center of the foam blocks so that it will not create a problem while shaping. Use toothpicks to temporarily hold the blocks in place. Drill two 1/8" cord holes in the outer edges of the end caps and put two lengths of heavy cord along the canoe sides to hold the ends in place. Put tension on the strings and allow the epoxy to cure.

When all sections are glued and the rough form of the canoe has taken shape, it is time to smooth the outside contour. This is done by carefully removing the excess foam with a wood rasp (handle removed). This is a messy job and a dust mask is necessary. Rubber gloves, a large fan, and a shop vacuum cleaner are handy for this step. The long axis of the rasp should be held parallel to the long axis of the canoe. The rasp should be moved in alternating 45 degree strokes. The shaping should be done until the desired contour is obtained. When completed, the canoe plug should look like the one in Photo 8 which is ready to be covered with fiberglass and epoxy.

INSTRUCTOR NOTES

Hot Wire Cutter

The items marked with an asterisk in the Equipment and Supply section are necessary to construct the hot wire cutter. Refer to Photos 5 and 10 for pictures of the wire cutter.

Start by boring two 11/16" holes across the 3 1/2" dimension of the 2" x 4". These holes should be centered across the 1 1/2" dimension and 1 1/2" from each end. Then drill a 1/4" hole 1/2" from one end of each piece of 11/16" conduit. Insert the pieces of conduit into the holes in the 2" x 4" with the 1/4" holes out. This should be

a snug fit, with the 1/4" holes aligned with each other.

Drill a small hole starting at the center of the 1/4" machine screw heads and angling out of the threaded portion of the screw just below the head. Mount the machine screws in the 1/4" conduit holes with a washer and a wing nut on the outside of the conduit. Tighten the nuts until two threads are exposed; then string the .041 stainless steel wire through the center holes in the machine screws. Secure the wire ends around the threads of the machine screws and then tension the wire by tightening the wing nuts.

Attach one wire of the heavy duty lamp cord to each piece of conduit by using sheet metal screws in a hole drilled 1/2" from the 2" x 4". Connect the other end of the lamp cord to the transformer output and the hot wire cutter is ready to use.

The transformer should be adjusted to between fifteen and eighteen volts, which will give enough heat to easily slice through the foam. Some trial cuts may be necessary to gauge the correct voltage and speed of cutting. The wire should be kept tight and never allowed to turn red. A SERIOUS BURN WILL OCCUR IF THE SKIN MAKES CONTACT WITH THE HOT WIRE.

Safe-T-Poxy

The resin matrix used in this experiment is Safe-T-Poxy, which is produced by the Hexcel Corporation. It was specially formulated to be low in toxicity and does not have the objectionable strong smell that other epoxies and polyesters have. It comes in kits with 7/8 gallon of resin and 2/5 gallon hardener (see Photo 9), and costs around sixty dollars per kit. When using a balance beam or small scale, mix the epoxy 100 parts resin to 44 parts hardener. An epoxy pump like the one in Photo 9 makes mixing easier, faster, and more convenient when large amounts are to be used. This dispenser meters the correct ratio of resin and hardener and costs around \$160 from one of the suppliers listed in the supply section.

The resin and hardener should be mixed in a waxless or plastic cup and then stirred thoroughly for about two minutes. There is a forty-five minute working time at 77 degrees F and a curing time of ten hours. The mixture may be brushed or poured on and then smoothed with a squeegee made from a plastic coffee can lid.

Future Applications

Template/hotwire cutting can be used to make rough mold cavities for small parts. Photo 10 illustrates the process for cutting a mold for a wind turbine blade tip. A small tip section was cut from the blade and secured to a two-inch block of foam. Using the air foil tip

section as a guide, a cavity was hotwired in the block of foam. A wire was placed around the airfoil before cutting to increase the size of the cavity which will allow defects caused by exotherm to be removed from the casting. After the cavity was cut, it was lined with duct tape and filled with a mixture of Safe-T-Poxy and microballons. The duct tape allows the cured casting to be easily removed from the mold as shown in Photo 11. After shaping, the blade tip looks like the one shown in Photo 12.

The products which can be formed by using template/hotwire cutting are limited only by one's imagination. With only slight modifications of the canoe experiment, a person can produce kayaks, racing shells, catamaran pontoons, small fishing boats, and snow sleds. Bodies for cars and recreational vehicles (go-carts, dune buggies, etc.) can also be formed by this process.

The lost foam process is an extension of template/hotwire cutting and works very well when making containers or cases. A gas tank can be formed by hotwiring a specially designed plug and then covering it with fiberglass and resin. When the skin is cured, a small opening can be made for the filler pipe and a solvent (lacquer thinner) poured into the foam causing it to dissolve. Cases are made the same way except they are sawed into two halves and the foam is removed by chipping. Examples of this process include racket cases for tennis/racketball, snow ski cases, battery boxes and tool cases for small power tools.

Conclusion

Template/hotwire cutting is not a process which demonstrates the latest in technical advancements. However, once a foam plug is cut, the learning process involved with covering it with fiberglass and resin teaches many of the important concepts used in the composite industry. This cutting process allows more freedom in design and reduces lead time. It can also be used as a substitute for the expensive presses and molds used to form products in industry.

The primary disadvantage of this cutting process is finishing the product after several layers of fiberglass cloth have been applied to the foam plug. Additional filling, sanding and painting operations are required to fill and smooth the fiberglass weave. This is not necessary on a molded product which has a built-in smooth painted outer surface.

The purpose of this experiment was to demonstrate the ease of forming composite products by hotwiring foam plugs from blocks of polystyrene. Because of the time restriction, it was not possible to explain the other steps involved in fabricating the complete canoe. A lab manual is being prepared which explains each step in making the canoe and gives many worthwhile pointers on using foam, reinforcing fibers, resins and related supplies.

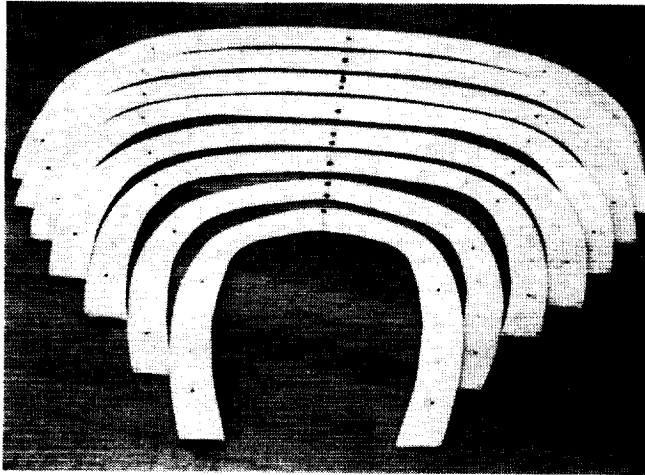
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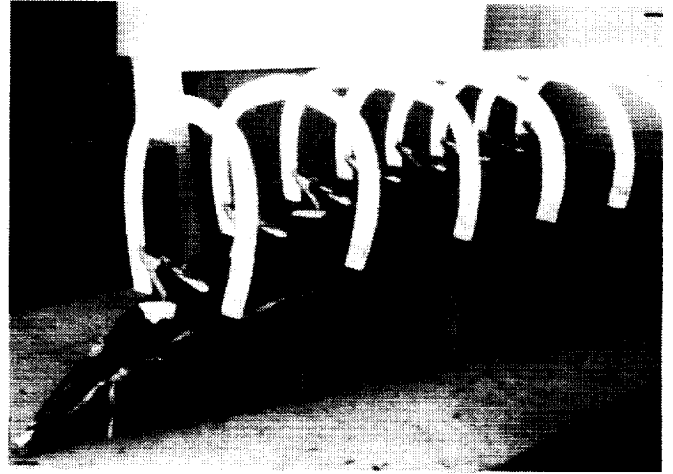
SOURCES OF SUPPLIES

1. Local builders supply (Builders Square, Payless Cashways, etc.)
polystyrene insulation board 2" x 2' x 8' \$5.00
required wood products
2. Local discount stores (Wal-Mart, K-Mart, etc.)
duct tape, mixing cups, dust masks, toothpicks, rubber cement,
lamp cord, chalk line cord, rubber gloves (pharmacy), mixing
sticks (pharmacy: tongue depressors), etc.
3. Aircraft Spruce and Specialty Co.
Box 424
Fullerton, CA 92632
714-870-7551
1-800-824-1930 (orders only)
epoxy, epoxy dispenser, fiberglass cloth, transformer, etc.
catalog - \$5.00
4. Wicks Aircraft Supply
410 Pine Street
Highland, IL 62249
618-654-7447
1-800-221-9425 (orders only)
(same supplies as Aircraft Spruce)
catalog - \$5.00
5. Advanced Plastics
11212 E. 112th Street N
Owasso, OK 74055
918-664-5410
resins, reinforcing cloth, etc.

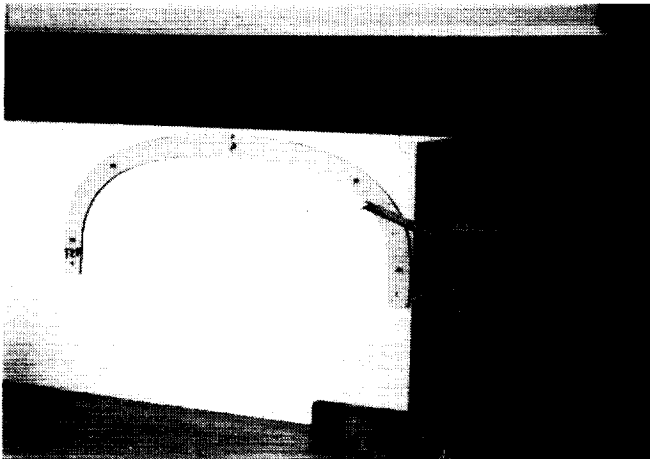
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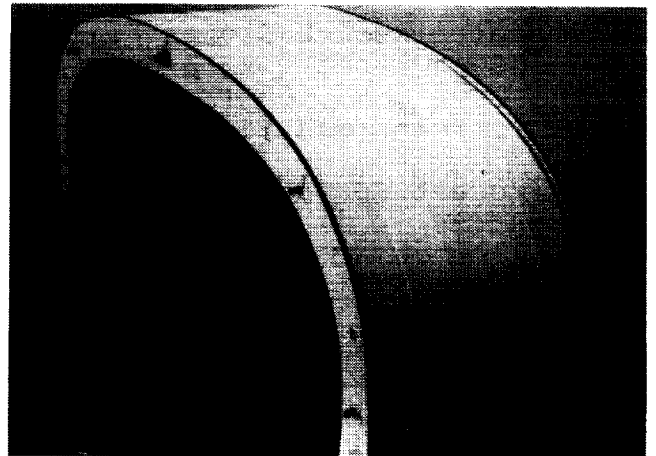
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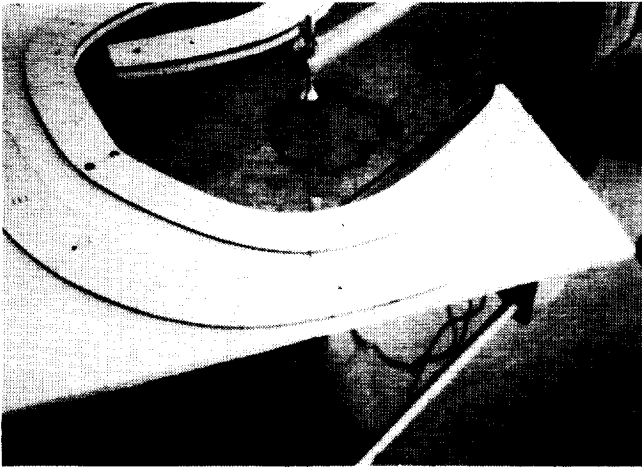
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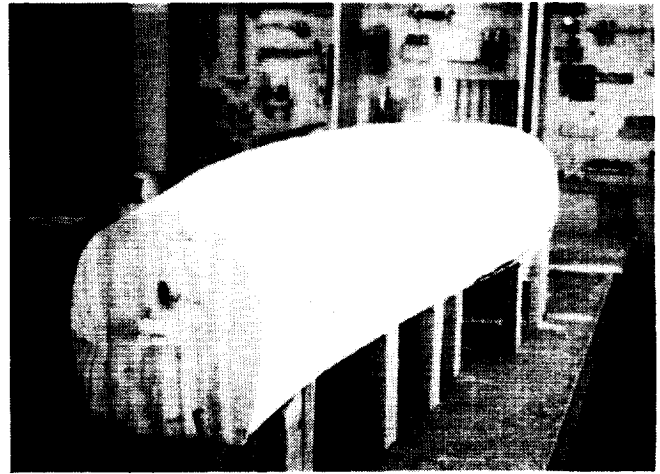
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PHOTOGRAPHS



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ORIGINAL PAGE
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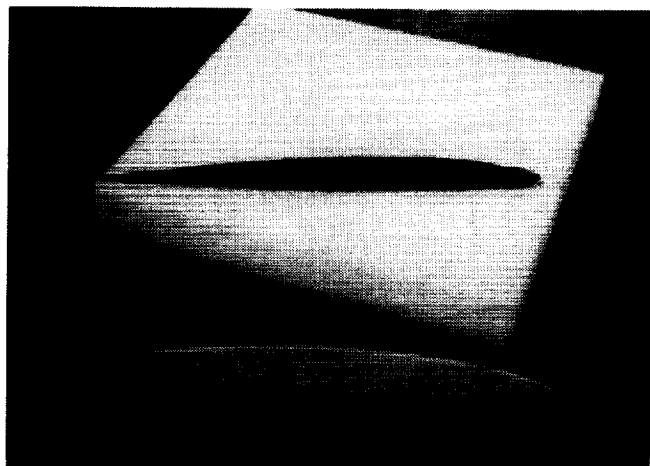
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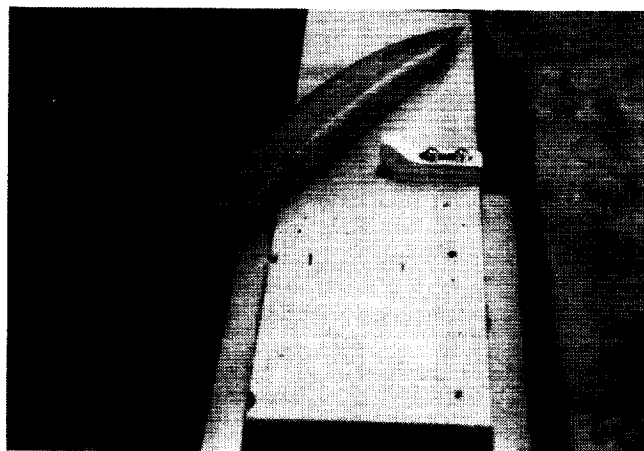
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12

ORIGINAL PAGE
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RUBBERLIKE ELASTICITY EXPERIMENT

Richard Greet and Robert Cobaugh
University of Southern Colorado

SUMMARY

Four rubber erasers from the campus bookstore, two green and two white, have been cut to have identical dimensions. These are loaded in compression with a mechanical test machine capable of applying cyclic loads and providing output to an X-Y plotter. The anelastic behavior of rubber as well as the relative stiffnesses of composite materials loaded transversely and longitudinally may be demonstrated to beginning students or used as input for computer analysis by advanced students.

DEMONSTRATION EXPERIMENTS

Anelasticity

In most instances, students are already familiar with the elastic behavior of metals--linear loading and unloading, without hysteresis loss. Figure 1 illustrates representative experimental behavior for the rubber erasers. Clearly seen is that the elastic deformation is highly non-linear with a large variation between loading and unloading consistent with the energy damping capacity of rubber materials. With the trade-mark identification still visible on the erasers, it is of interest to the students that such familiar everyday materials can behave in an unexpected way.

Relative Stiffness

Central to any mechanical testing program is to compare one material to another. This can be demonstrated to students by producing curves for the green and the white erasers on the same sheet of graph paper. Because the samples have been cut to have identical geometries, the comparison is immediate without the necessity of computations.

Composite Materials

Prototype composite materials can be constructed by stacking the erasers in different ways as shown in Figure 2. Compression of these stacked erasers demonstrates the different stiffness of composite materials loaded longitudinally and transversely. Again because of the identical geometry, the comparison is immediate, without computations.

QUANTITATIVE POSSIBILITIES

Data Generation

As a start to quantitative analysis, students can measure the dimensions of the samples and convert the various FORCE-DEFLECTION graphs obtained into STRESS-STRAIN curves. In the analysis mentioned below, individual points from the curves are required. These may be read off manually by the students, or two alternatives can be used to emphasize the engineer's use of computers in today's world. In one alternative, the graphs are put onto a digitizing pad and a mouse or cursor is used to generate numerical points directly into a micro-computer. In the other alternative, the machine output that drives the X-Y plotter is fed to a data collection device that is programmed to sample the voltages while the experiment is being run. The data sampling device has internal memory and can be subsequently connected to a microcomputer to transfer data.

Elastic Modulus

In addition to being a function of the strain, the elastic modulus of rubbery materials is several orders of magnitude lower than that of metals. To obtain numerical modulus values, students are directed to generate the stress-strain data along the center of the hysteresis loop. These points are then used with a least squares fit computer program to generate the coefficients of a quadratic or higher order fitted polynomial. The derivative of the polynomial is then interpreted as the instantaneous modulus during the experiment.

Damping Capacity

The area of the hysteresis loop represents the energy absorbed by the sample per unit volume per cycle of loading. To obtain this area, students must generate stress-strain numerical values for the loop itself. These numbers are then used with a computer program that performs Simpson's one third rule numerical integration to compute the area.

CONCLUDING REMARKS

The experiments described can address students at many levels. Without numerical computations, the experiments demonstrate qualitatively several aspects of materials behavior. For analysis, the computer is used and the analytical techniques themselves might be the emphasis of an instructor. Still another focus could be the data collection system itself--data sampling and/or real time analysis.

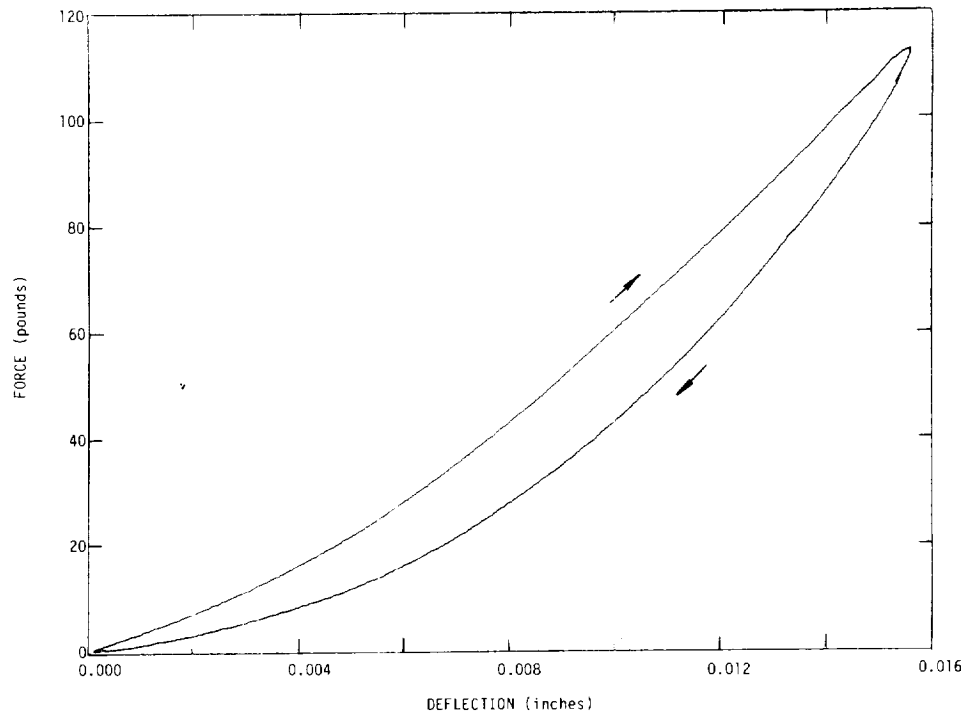


Figure 1: (Force) versus (Deflection) for rubber eraser undergoing cyclic compression.

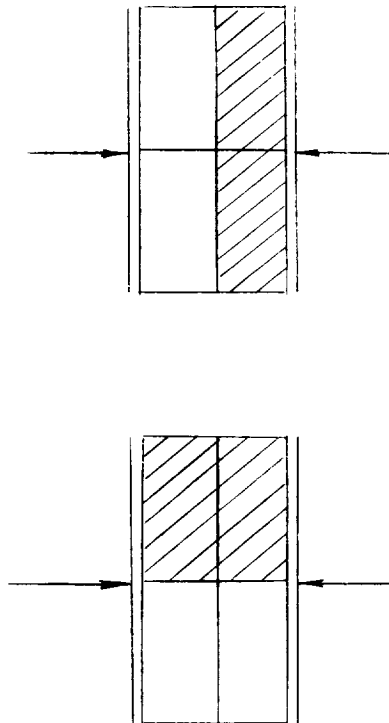


Figure 2: Stacking of erasers to simulate composite materials.

THE EFFECT OF THERMAL DAMAGE ON THE
MECHANICAL PROPERTIES OF POLYMER REGRINDS

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SUMMARY

Reprocessed polymers are subjected to high processing temperatures that result in the breakdown of molecular chains and changes in the molecular structures. These phenomena reflect in the mechanical properties of materials. Practically every regrind is seen as a new material.

These experiments deal with the molding, regrinding, and reprocessing of test specimens for the study of their mechanical properties. The comparative test data from each recycled material would give students an insight of the molecular structures and property degradation.

Three important rheological and mechanical properties such as melt flow, impact strength, and flexural strength are to be determined. These properties play key roles in the selection of engineering materials.

The material selected for demonstration was Makrolon 3000L, a polycarbonate thermoplastic from Bayer AG. The thermal degradation due to repeated processing reflects in the decrease in molecular weight and breakdown of molecular chains causing increase in melt flow. The Izod-impact resistance and the flexural strength deteriorate gradually.

INTRODUCTION

The demand for plastics has increased tremendously over the last decade. More than half of the demand for polycarbonate goes into communications and electronics. As demand increases in the more critical engineering applications, the use of all available feed stock, even regrind, becomes a necessary requirement.

This work investigates the effect of reprocessing on the mechanical properties of polycarbonate. The polycarbonate material studied was Makrolon 3000L manufactured by Bayer AG.

Polymers are exposed to heat and mechanical shearing during injection molding, which results in the breakdown of molecular

chains. Practically every regrind is seen as a new material. The author suggests that the students should mold their own specimens and regrind and reprocess the same material using appropriate processing parameters. The comparative test data from each recycled material give students an insight of the molecular structures and corresponding property degradation. Three important mechanical and rheological properties, such as melt flow, impact strength, and flexural strength can be easily determined and play key roles in the selection of engineering materials.

TESTING OF MECHANICAL PROPERTIES

Equipment

Injection molding machine, scrap grinder, flexural stress tester, impact tester, and melt flow measuring instrument.

Test Specimens

Test specimens were prepared according to DIN 53452 for flexural stress and according to DIN 53453 for izod impact tests.

50mm x 6mm x 4mm test specimens were injection molded. The specimens were molded at 310 C with a cycle time of 40 seconds. Specimens for impact tests had a notch 0.9 mm wide and 1.3 mm deep.

To start 300 specimens were molded, tested, and reground. Then 240 specimens were molded with the same processing parameters, tested, and reground. The third, fourth, and the fifth molding followed.

Test procedure

For every processing ten specimens were tested for flexural stress, twenty specimens for impact strength, and five readings were taken for melt flow readings.

Flexural stress

The specimens were placed on supports at both ends and load applied at the center until failure. Figure 2 shows the testing apparatus manufactured by Zwick. Bending stress was calculated using the applied load, the bending moment, and the moment of inertia of the sample.

Flexural stress $\sigma_b = M/(I/c)$ (MPa)
 $I/c = bh^2/6$

b=width of the specimen
h=height of the specimen

M=bending moment= PL/4
P=load at the center

Notched Izod impact test

Izod impact tests were done on an impact tester from Zwick. The energy required to break a notched sample with respect to the area under the notch is considered as impact strength. The test is done by clamping a specimen in the base of a pendulum testing machine. The pendulum is released and the energy consumed in breaking the sample was recorded. The impact strength was calculated by using the energy as recorded by the tester and the cross sectional area of the sample.

Impact strength $\alpha = E/(bh_k)$ (kJ/m²)

E=energy reqd. to break the sample
b=width of the sample
h=height of the sample

Melt flow

The melt flow test indicates relative flowability of polymers in the melt form. It is the most basic test on thermoplastic polymers.

Melt flow of a polymer is determined by the amount of viscous polymer forced through a standard orifice at a certain temperature under load for a certain time. The melt flow apparatus is shown in figure 3.

RESULTS AND DISCUSSION

A set of sample test results for only the first moldings are given at the end of this paper. Table 1 shows the results on flexural tests, table 2 shows the results of notched izod impact tests, and the results of melt flow test are given in table 3. Finally a summary of the test results for all five moldings is given in table 4 which is also graphically reproduced in figure 4.

Flexural Stress

Bending stress, a form of fatigue, is a unique stress with characteristic mechanisms that are distinctly different from those of static or impact stresses. Bending stress data are helpful in understanding plastic fatigue performance, ranking materials, and qualitatively guiding design.

The data on bending stresses shows a wide variety of results. The Makrolon 3000L molded at 310 C remained relatively the same until the third molding in which a slight increase in resistance was observed, after which a significant decrease occurred. This may be explained by a difference in fiber orientations, or a difference in melt flow or significant decrease in the molecular weight.

Notched Izod impact test

During impact testing, the specimens undergo three stages of development. The elastic stage, then the crack propagation stage and finally the separation stage. As the specimens are remolded, a significant decrease in impact resistance is observed. As the material is recycled, its thermal history is changed as well as the flow patterns.

Melt Flow

The melt flow increases with each cycle because of main chain scission. The Makrolon 3000L experienced a significant increase in melt flow between the first and the fourth moldings but basically leveled off at the fifth. This is the result of the high processing temperature which causes a decrease in molecular weight and a breaking down of the materials' main chains.

TABLE 1
Flexural Test
Makrolon 3000L
First Molding

Specimen	Load (N)
1	151.76
2	154.02
3	152.06
4	152.06
5	156.96
6	153.04
7	151.56
8	157.94
9	155.98
10	151.56

TABLE 3
Melt Flow Test
Makrolon 3000L
First Molding

Time (min)	Weight (g)
0	-
2	0.5925
4	0.6330
6	0.6607
8	0.6615
10	0.6305

Melt Index = 3.185/10min

TABLE 2
Notched Izod Impact Test
Makrolon 3000L
First Molding

Specimen	Energy (J)
1	0.761
2	0.790
3	0.682
4	0.775
5	0.741
6	0.834
7	0.780
8	0.736
9	0.510
10	0.804
11	0.844
12	0.530
13	0.804
14	0.824
15	0.569
16	0.608
17	0.765
18	0.716
19	0.608
20	0.824

TABLE 4
SUMMARY OF TEST RESULTS

Temperature 310° C	Molding	Impact Strength kJ/m ²	Bending Stress MPa	Melt Flow g/10min
Makrolon 3000L	1	44.83	96.82	3.18
	2	32.08	95.94	3.64
	3	15.79	98.30	6.65
	4	4.02	broken	12.33
	5	4.02	broken	12.33

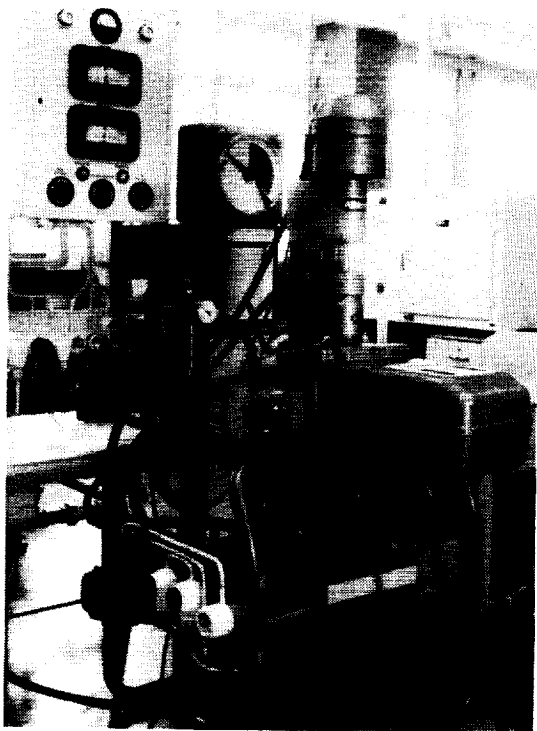


Figure 1. Injection molding machine

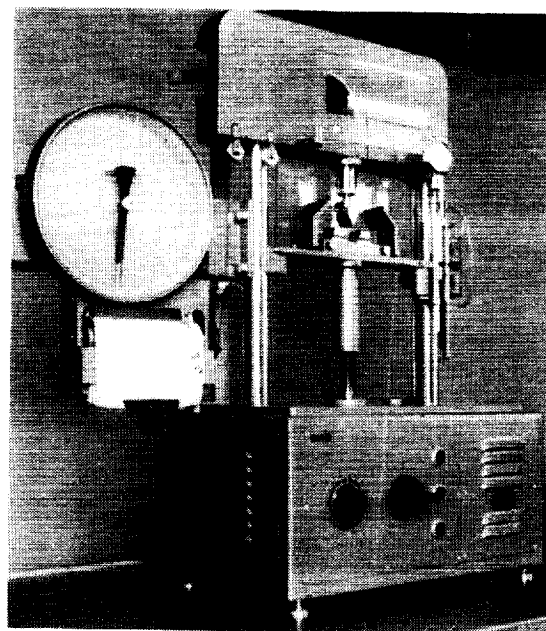


Figure 2. Flexural stress tester

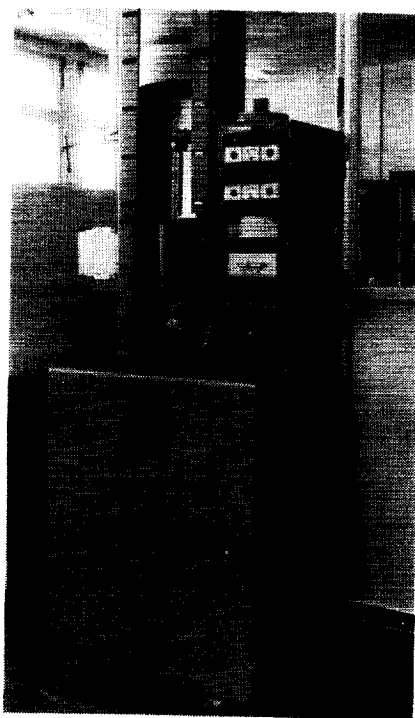


Figure 3. Melt flow apparatus

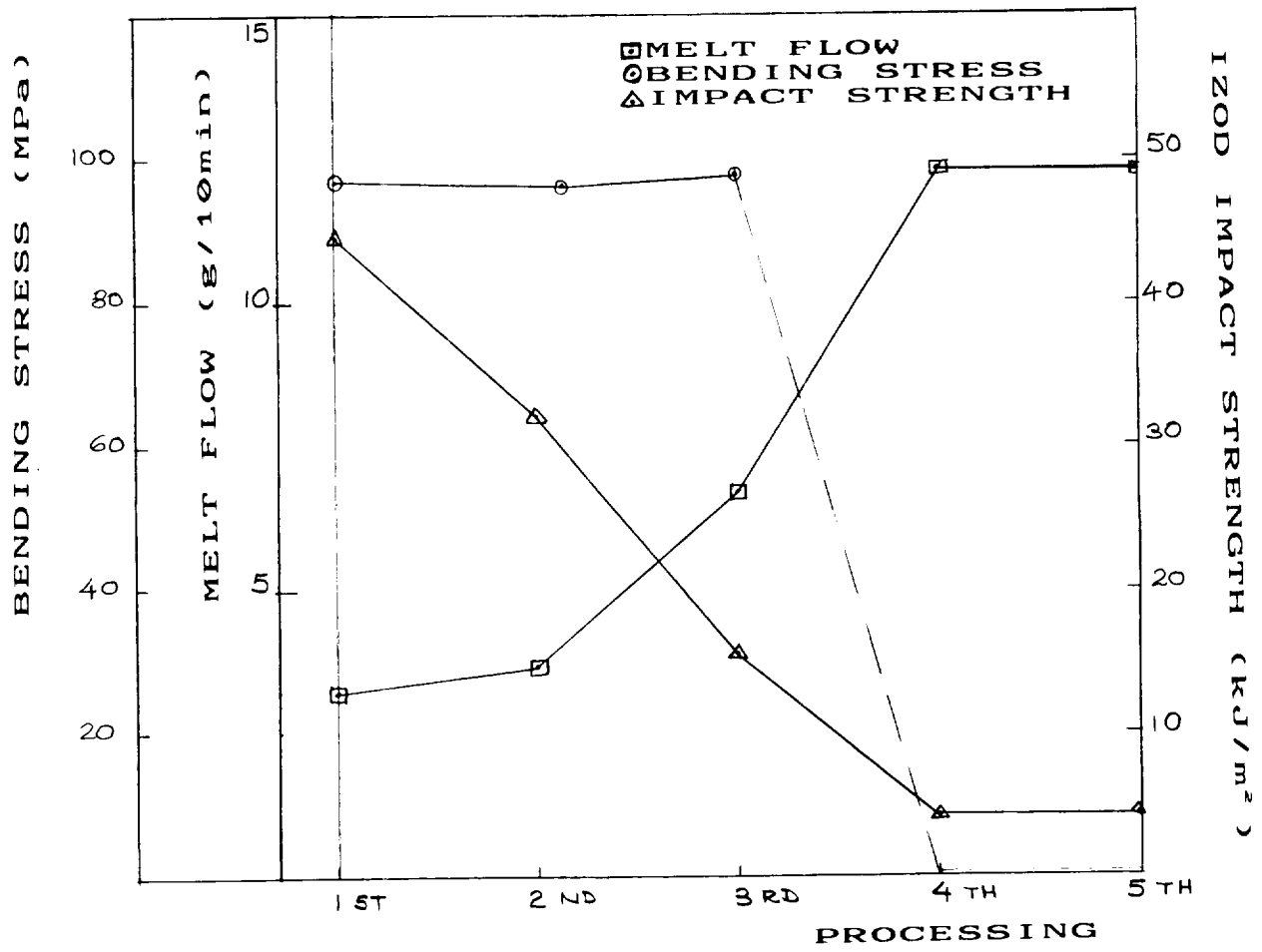


FIGURE 4

EXPERIMENT TITLE: Demo of Three Ways To Use a Computer to Assist in Lab

AUTHOR: J. P. Neville

AFFILIATION: Wentworth Institute of Technology, Boston, Mass.

Key Words:

- 1.) Enhancement of video presentations
- 2.) Remedial help and testing
- 3.) Information source and lab simulation

PREREQUISITE KNOWLEDGE REQUIRED: None

OBJECTIVE:

Help the slow learner and students with a language problem, or challenge the advanced student.

EQUIPMENT and SUPPLIES:

Computer capable of generating movies

PROCEDURE:

1.) Technology has advanced to the point where images generated on a computer can easily be recorded on a VCR and used as a video tutorial. This transfer can be as simple as pointing a video camera at the screen and recording the image. For more clarity and professional results, a board may be inserted into a computer which will convert the signals directly to the TV standard. Using a computer program that generates movies one can animate various principles which would normally be impossible to show or would require time-lapse photography. For example, you might show the change in shape of grains as a piece of metal is cold worked and then show the recrystallization and grain growth as heat is applied. More imaginative titles and graphics are also possible using this technique.

2.) Remedial help may also be offered via computer to those who find a specific concept difficult. A printout of specific data, details of the theory or equipment set-up can be offered. Programs are now available that will help as well as test the student in specific areas so that a "Keller " type approach can be used with each student to insure each knows the subject before going on to the next topic.

3.) A computer can serve as an information source and contain the microstructures, physical data and availability of each material tested in the lab. With this source present "unknowns" can be evaluated and various tests simulated to create a simple or complex "case study" lab assignment.

REFERENCES:

In-Office Video Taping: What will be Next?, by Prof. John Lindenlaub of Purdue, Session 2630 of the 1987 ASEE Annual Conf., Proceedings Vol. 3 page 1337
Multimedia Software Development for Electronics Technology Students by Dr. Charles Schuler, Technical Education News (Spring 1989)
Hypermedia by Dawn Stover, Popular Science, May 1989

Equipment available in video stores in 1989:

Camera: There are many available - both from a tape size and feature point of view. There are also industrial versions as well as those designed for home use. Prices also run the full range with \$1000 being the tag on one that does almost everything.

Features needed:

The camera needed for making tutorials need not be capable of recording but it should have a zoom lens. If it can record, it may take the place of one of the VCR units shown in the diagram.

The camera will need to be capable of being operated at a distance. If it comes with a remote control it will be easier than having the shop rewire the unit. It should have an electrically operated Zoom as this is the major control that you will want to change during taping.

You can add simple titles with many cameras and this may be the least expensive way to superimpose them on your finished product.

Fade capability is a nice feature but may not see much use, depending on your style.

VCR: "Flying erase heads" are needed if a smooth transition is to be made from one scene to the next. This usually means you will need a machine with 4 heads.

The ability to change the speed of the picture is a good feature since you can play back at a different speed than recorded and use this new speed in your final edited tape. Increasing the speed of animation this way may help it run smoother. Pictures taken off a computer can also be enhanced this way.

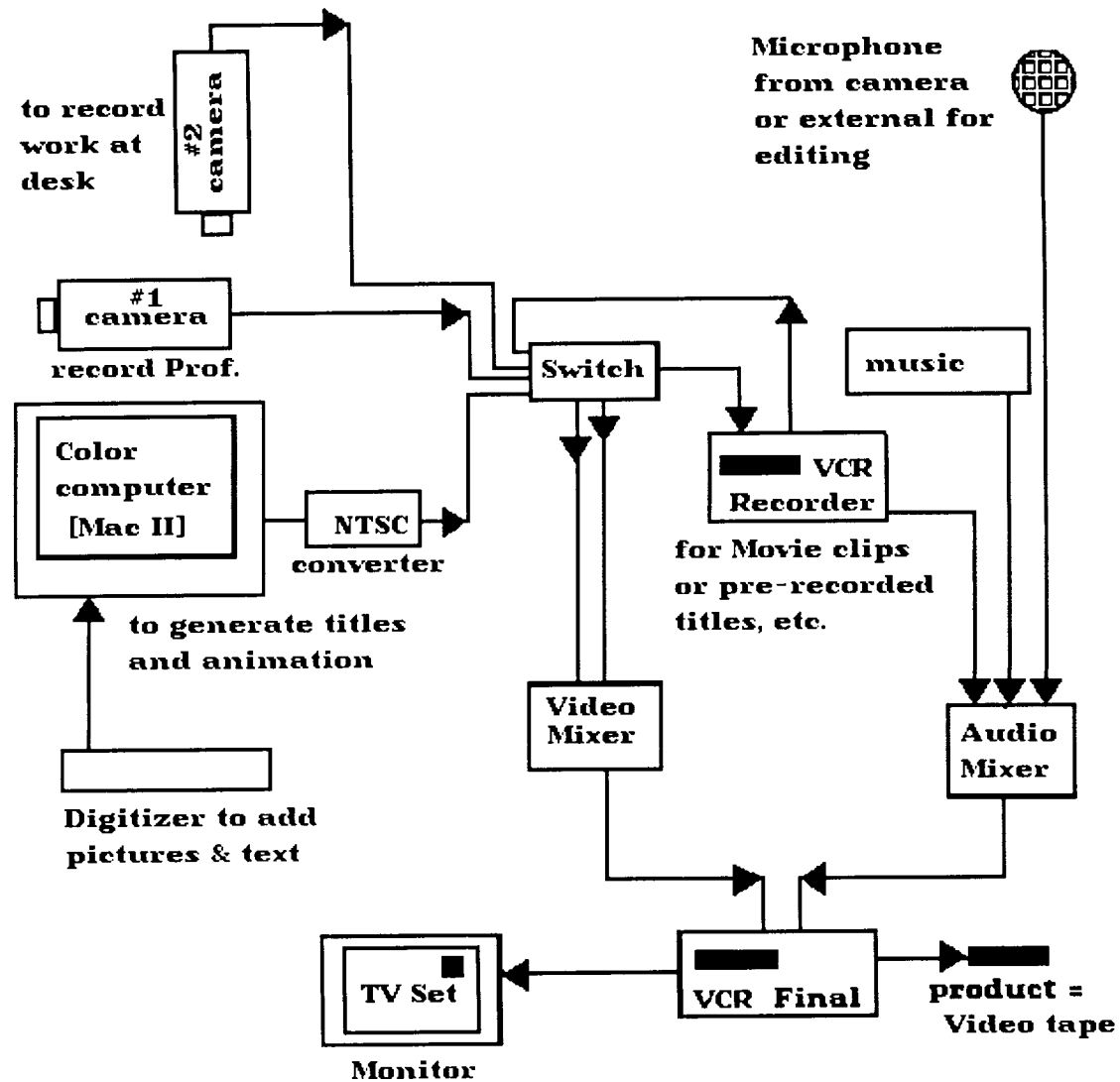
Editing Equipment:

Sound mixing: To add music or voice over your original tape and still keep the original sound track you will need a **Video Tape Audio Mixer**. Sima Products Corp. of Chicago is currently marketing a "SoundMixer" in department stores for around \$70.00. This unit is capable of mixing 3 inputs, changing the volume of each separately. This allows you to use PIP [picture-in-a-picture] and have the voice come from one source.

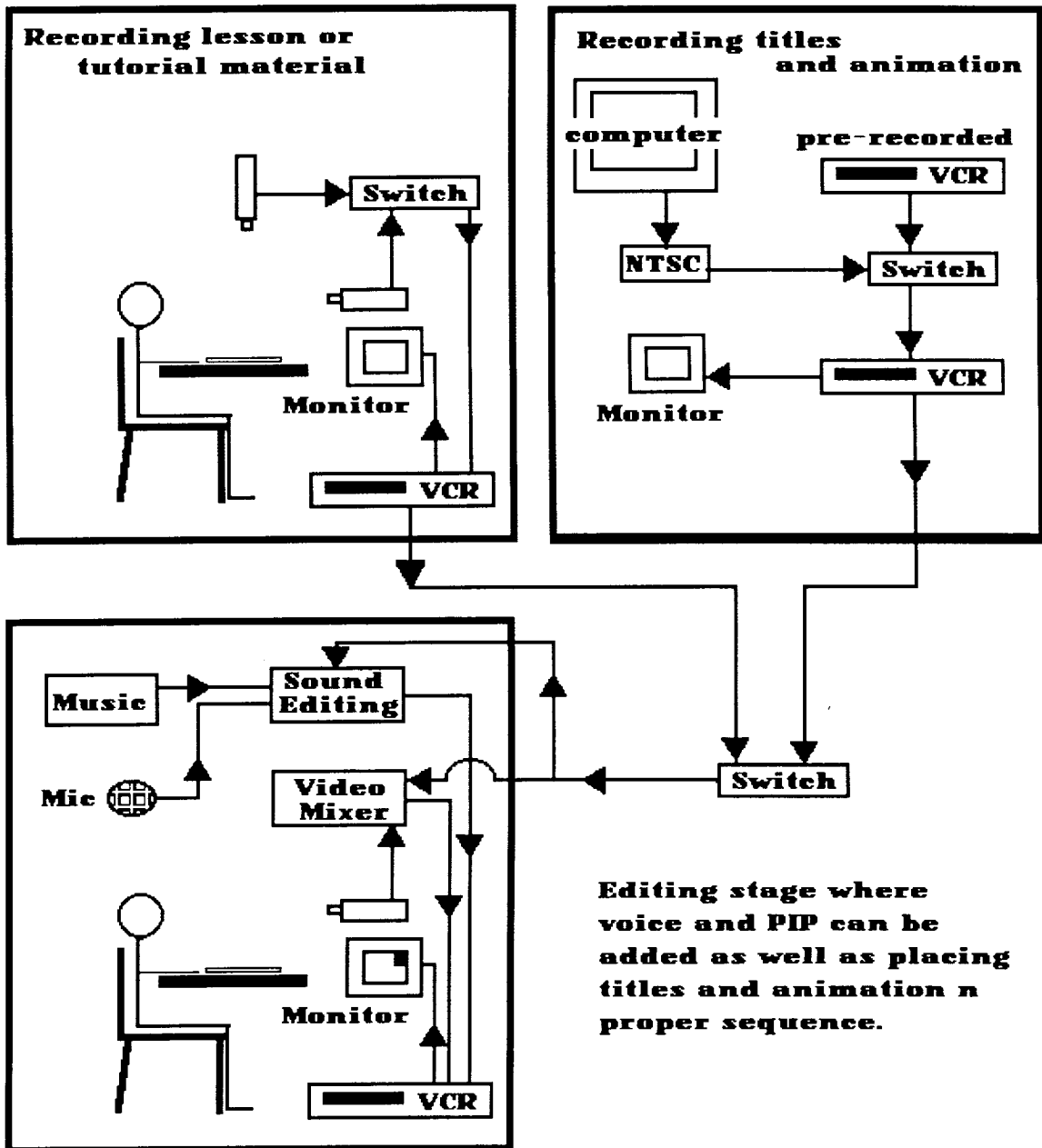
Picture-in-a-Picture: Rabbit Systems of Santa Monica, Ca. market a "Double Play" unit for \$250.00 which is designed to show two TV stations on your screen at once, [PIP]. It is a rather useless, overpriced unit as advertised but makes a great, inexpensive way to add yourself to a tutorial. Setting the unit to Ch. 3 one can mix a live camera and a tape and send the result to another VCR. This will create a "Talking Head" in any one of the four corners of the picture. The insert can be added or removed at any time and you can swap the small picture for the large at any time also. It is a nice way to add a personal touch to film clips or animation. It is also a way to show a problem and do it at the same time in a tutorial.

DirectEd is a unit available from Videonics of Campbell, CA. for around \$500 which generates titles in various colors and provides several different transition modes from one scene to the next. It also provides a few "graphics" you can add. Once you have marked your tape, the machine will automatically use your original to generate a new tape, removing parts or inserting the titles you desire in the proper places. It is much less expensive and less work than using a computer, but it provides no animation.

SET-UP USED TO MAKE VIDEO TUTORIALS



THREE STEPS OF THE PROCEDURE



The Magnetization Process - Hysteresis

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The way in which a piece of magnetic material behaves when in a changing magnetic field tells a great deal about its properties. The magnetic properties of any material are essentially a superposition of the magnetic properties of a larger number of magnetic regions called "domains". A domain is a region in a magnetic material where the elementary atomic magnetic moments point in the same direction. Between domains are transition regions called "domain walls" in which the atomic magnetic moments change directions from one orientation to another.

Suppose we put a piece of iron in a long coil where the field produced by the coil, H_{app} is uniform. With no field in the coil, the iron breaks up into many alternating domains with equal volumes of up and down domains. If we apply a current to the coil, the domain walls will start to respond as soon as the field exceeds some minimum strength called the coercive field. If we make a graph of the net magnetization of the iron, M , (on the vertical axis) as a function of the field intensity, H , produced by the current in the coil, we obtain a curve such as that shown in Fig. 1.

As the current in the coil (and its magnetic field) increase, the up domains grow larger and larger as the down domains decrease and the net magnetization increases. At some field level all of the down domains have been eliminated, and the material is said to be **saturated**. We designate the field H_{sat} . Fields above H_{sat} no longer change the net magnetization. This is just what we should expect. When all the magnetic moments are lined up with the field, the net magnetization cannot increase further, no matter how much current is applied.

If we now decrease the current, the net magnetization will decrease as well, but it does not begin to decrease until the external field, H_{app} is somewhat below the saturation level. Then, down domains appear, gradually grow and the net magnetization decreases. Notice that the graph does not retrace the same curve. Even when the current is back to zero, there is usually some magnetization left. This is called the **remanent magnetization**.

Increasing the current again, but in the opposite direction gives us an opposite field, $-H_{app}$. The magnetization continues to decrease and when H_{app} becomes equal to $-H_C$, the coercive field, the magnetization reaches zero again. Further increasing of the negative field causes the magnetization to increase in the opposite direction until the sample is again saturated when the field reaches $-H_{sat}$. On decreasing the strength of $-H_{app}$, the negative field, up domains reappear, and eventually at H_C the net magnetization is brought back to zero.

If we continuously change the current back and forth, that is, if we applied alternating current to the coil, a loop is traced out.

One of the outstanding features of this curve is that there is a difference in the path of the magnetization for increasing and decreasing field. This is called **hysteresis** and the curve is called a **hysteresis loop**. Hysteresis is seen in many other physical systems. For instance, suppose we had a block of wood resting on a horizontal sheet of sandpaper and held in a certain position by springs. If we plotted the position of the block against an applied force that went alternately positive and negative, the plot would look very much like the magnetic hysteresis loop, except that it would not show saturation. In both the mechanical and the magnetic cases, as you go around the hysteresis loop energy is dissipated. It turns out that the area of the loop is directly proportional to the energy dissipated and therefore, to the coercive field. In transformer applications it is desirable to minimize the magnetic energy losses. Thus, one looks for magnetic materials with the smallest H_C .

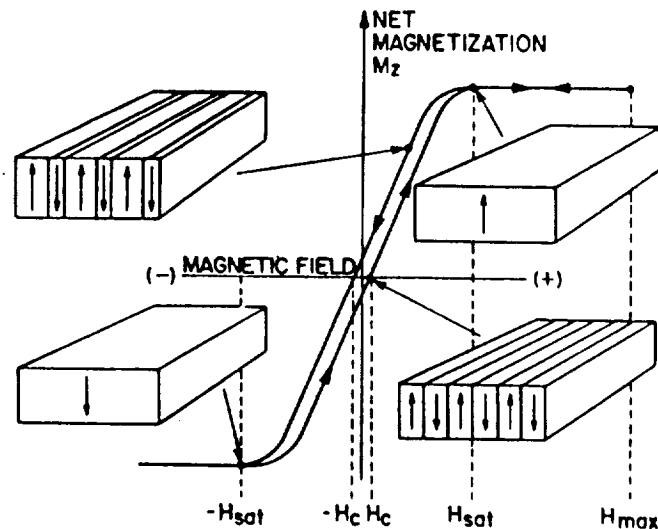


Fig. 1- Sketch of a magnetic hysteresis loop and the corresponding domain structures of a plate with easy axis perpendicular to the surface.

Magnetic materials in which the domain walls move very easily, and which therefore have low coercivity, low remanence, and very narrow hysteresis loops, are called **soft magnetic materials**. The use of the adjective "soft" arose because these tend to be mechanically soft and easily deformable. An iron nail which has a relatively thin hysteresis loop will bend. Over the decades there has been a great sustained effort to improve the magnetic properties of this class of materials.

At the other extreme are magnetic materials which we want to stay magnetized, so called permanent magnets. These we want to have as large a magnetic moment as possible, and when magnetized, to retain that moment even when exposed to moderate magnetic fields. These are called **hard magnetic materials**. These materials have high coercivity and remanence and thus wide hysteresis loops. Typically they are mechanically hard, often exceedingly brittle. A permanent magnet made of Alnico will break quite easily. Hard magnetic materials can be prepared by introducing non-magnetic impurities which hinder the motion of domain walls. Another way to make a hard magnetic material is to divide it into particles so small that a domain wall will not fit within the particle. If there can be no domain wall, the magnetization cannot reverse by the domain wall mechanism we have described. Only when we apply a field which exceeds the anisotropy will the magnetization flip over. Thus a further requirement is for a large anisotropy. The magnetic recording materials used in tapes and credit cards have been developed using this approach.

The achievement of good hard magnetic materials also has important economic consequences. Very recently the discovery of greatly improved hard magnetic materials has revolutionized the design of small permanent magnet motors. The development of materials is a vital part of the ongoing improvements in magnetic recording on which the technology of our society is evermore dependent.

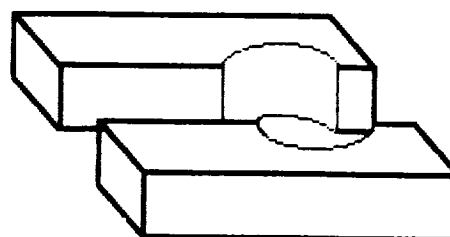
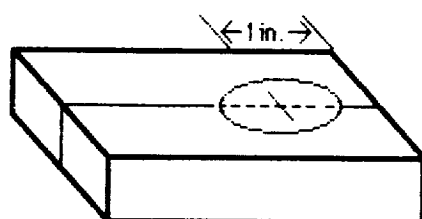
An apparatus and activity which show hysteresis in materials in a qualitative manner have been developed. The activity and construction notes follow.

The hysteresis loops introduced above constitute a tremendously valuable tool in characterizing magnetic materials. In transformers and motors the magnetization is swept through a hysteresis loop 60 times every second. Each time some of the electrical energy is converted to heat. If the coercive force is large, the hysteresis loop is fat. Consider a practical application of this principle. A material with a large coercive force in addition to the eddy current losses, would cause a motor to become very hot. Materials with a large coercive force, require large expensive motors. On the other hand if the domain walls move in very small fields, i.e., if H_C is tiny, motors can be smaller and less electrical energy is wasted in heating up the atmosphere.

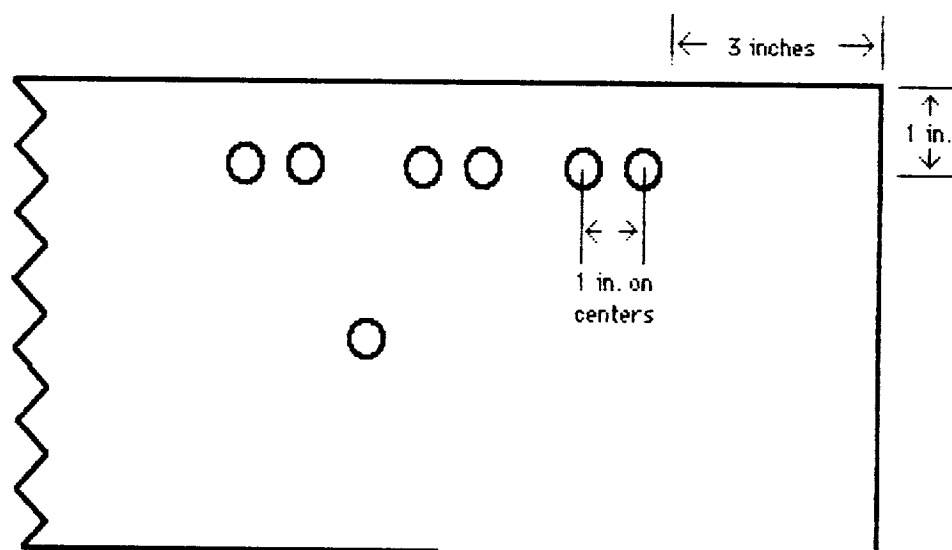
Construction Notes: Hysteresis

1. You can build a stand for the coils using one foot of 1x3 pine, two 3 - 1/2 inch pieces of 2x2 pine, and a 3 - 1/2 inch piece of 1x2 pine.

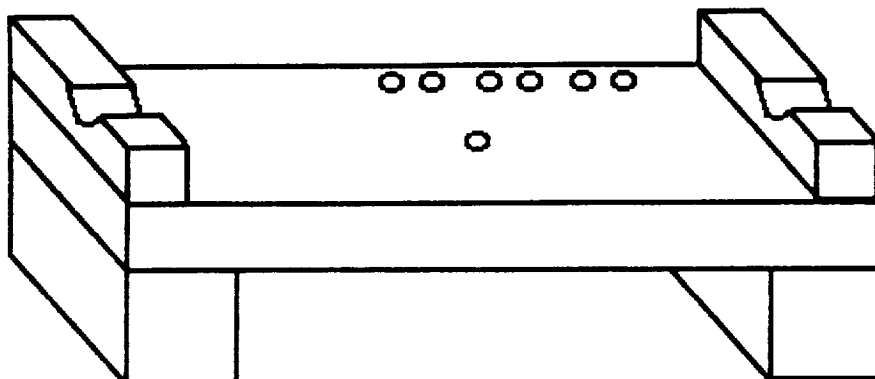
2. Drill a 3/4 inch hole in the center of the piece of 1x2, one inch from one end as shown. Then cut the piece of 1x2 lengthwise down the center.



3. Drill three sets of two 5/16 inch holes on the 1x4 three inches from one end and one inch in from the edge as indicated below. Use a 1/2 inch bit to countersink these six holes to a depth of 1/2 inch. This is necessary so that the binding post will fit. Drill another 5/16 inch hole on the center of the 1x3 board.

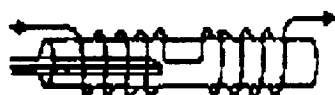


4. Now glue the two pieces of 2x2 on the bottom ends and the two pieces with the half circles and the top ends. The completed stand should look like this:

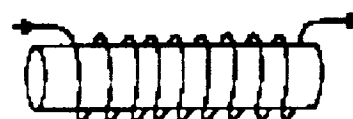


5. Take one foot of 3/4 inch outside diameter tubing. Leaving about 1 - 1/2 feet of loose wire and starting one inch in from the left hand end, wind about 300 turns clockwise of #28 enameled magnet wire. After this leave a one inch length of straight wire. Hold it in place with a piece of tape and then wind 300 turns counterclockwise, continuing to move to the right end of the tube as shown below. Leave another 1 - 1/2 feet of loose wire on this end. Hold the turns in place with tape.

6. Take six inches of one inch inside diameter tubing. Leaving 1 - 1/2 feet of wire loose on each end, wind about 275 turns clockwise on this tube. Hold the turns in place with tape.



SMALLER TUBE



LARGER TUBE

7. Now slide the larger diameter tube over the smaller, covering the coils. Label all four wires as to which coil they are from: the outer, larger diameter, coil or the inner, smaller diameter, coil. Thread all four ends through the hole on the middle of the stand.

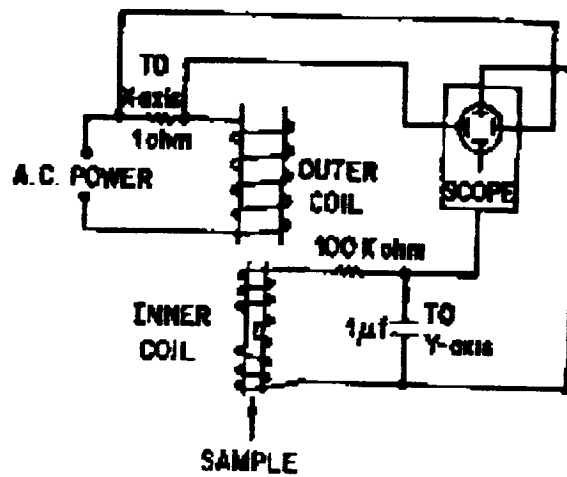
8. Tape the ends of the smaller tube into the half circles in the wood on either end of the stand and turn the stand over.

9. Place three sets of binding posts in the six holes and tighten the nuts. Label the three sets of posts as "AC IN", "X- axis", and "Y - axis".

10. Identify the two ends of the outer, larger diameter, coil. Solder a one ohm, three watt resistor to one of these ends. Solder the resistor to one of the binding posts of the set marked "AC IN". Solder the other end to the other post of this set.

11. Identify one of the ends of the inner, smaller diameter, coil and solder a 100 K ohm resistor to it. Then solder the 100 K ohm resistor to one of the posts labeled "Y - axis". Solder the other end of the inner coil to the other post of this set. Finally solder a 1 μ f capacitor to both posts of this set.

12. Solder a wire from the post that has the one ohm, three watt resistor to one of the posts labeled "X - axis". Solder another wire from the joint between the one ohm resistor and the end of the outer coil to the other post labeled "X - axis". Check your circuit with the diagram below

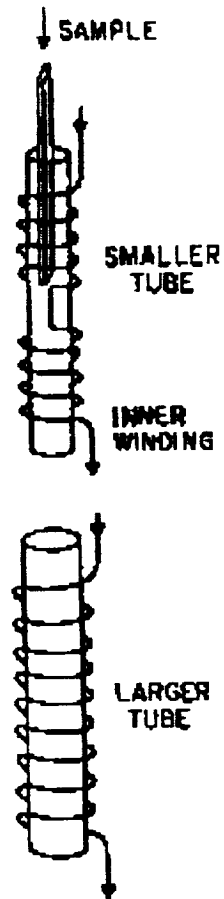


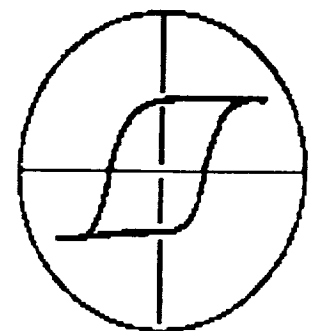
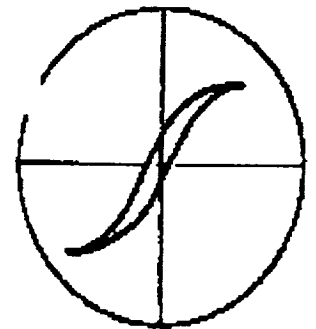
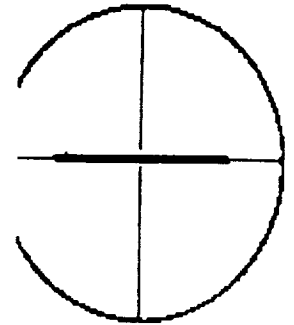
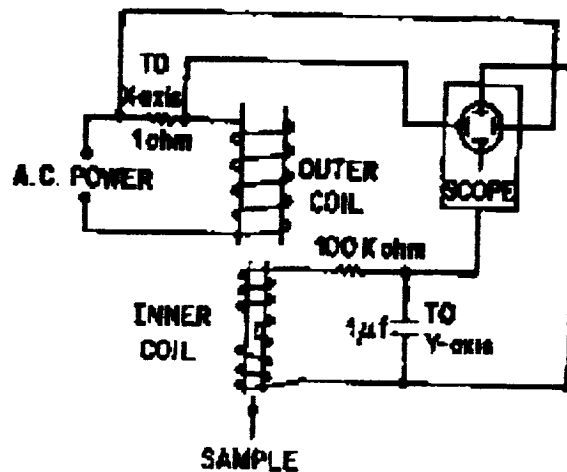
Activity: Hysteresis of Common Magnetic Materials

The way a material is magnetized in the presence of an alternating magnetic field can be displayed on an oscilloscope. The resulting "hysteresis loop" reveals important properties of the material.

You will need: an AC power supply (5 to 10 volts), an oscilloscope, and several samples of different magnetic materials.

1. When current is passed through the outer coil, it acts as the primary of a transformer, producing a magnetic field along the length of the tube. The inner coil acts as the secondary of the transformer and senses the field produced by the primary or drive coil. The two oppositely wound halves of the sense coil give equal and opposite currents which cancel each other perfectly. When a magnetic sample is placed into the tube within the first half of the sense coil, the two output signals of the secondary coil halves no longer cancel and the result depends on the extra field produced by the sample itself.
2. Pass a 60 Hz alternating current (about 0.5 to 1.0 amps) through the outer primary coil using the power supply (or a six volt step-down transformer plugged into a wall socket) with a one ohm resistor in series. The voltage across this series resistor is the X-input to the oscilloscope. It is numerically equal to the current in the coil, which is proportional to the field in the center of the coil. The Y-input to the oscilloscope is the signal from the sense coil attached as shown in the figure. A resistor and capacitor in this sensing circuit are used to filter out extraneous high-frequency signals. On the oscilloscope, set the Y-axis scale to 10 mV/cm and the X-axis scale to 0.2 V/cm.





- With no sample in the coil, the output should be a horizontal straight line. (If the coils are not well matched, this line will slope up or down. Sliding the outer coil back and forth over the inner coil should result in a better balance and a better horizontal line.)
- Place various metallic objects into the first half of the sense coil. Non-magnetic samples such as an aluminum nail will have hardly any effect on the output. A hacksaw blade will produce an open hysteresis loop. Paper clips, a screw driver, a coat hanger, transformer core material, will each produce an interesting loop. (Adjust the Y-axis scale to display the best loop for each sample.) Note that some loops are thin and some are wider or square. Using a small piece of silicon iron, or several together, see the difference in the loops when they are magnetized along the rolling direction or perpendicular to it. Try a large iron nail and see the changes produced by flattening it with a hammer and by heating it in a Bunsen burner. Explain the shape of each loop in terms of the motion of domain walls and the rotation of the magnetization within each sample.

- The magnitude of the applied field can be calculated from the formula:

$$H = (4 \pi N I) / 1000 \times l$$

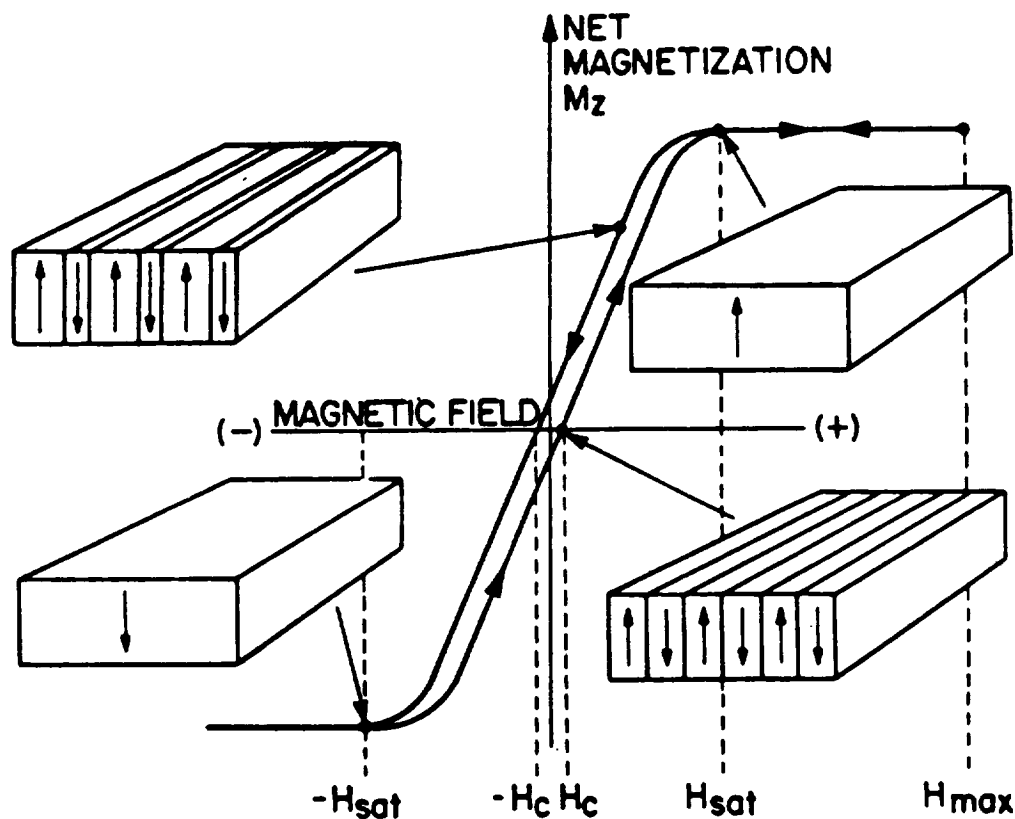
where N = number of turns, I = current in amperes, and l = length of the coil in meters. If the circuit has been designed so that $I = 1$ amp, $N = 1000$ turns and $l = 12.5$ cm, then the field in the center of the coil is

$$H = (4 \times 3.1416 \times 1000 \times 1) / (1000 \times 0.125)$$

$$= 1.005 \text{ amps/meter} = 80 \text{ oersteds}$$

(Remember that the earth's magnetic field is about 1 oersted.)

If the X-axis scale on the oscilloscope is set at 0.2 V/cm, and the voltage across a 1 ohm series resistor is used as the input, each cm on the horizontal axis is equivalent to 16 oersteds. This calibration can be used to estimate the value of the coercive force from the half-width of the hysteresis loop at the X-axis. The Y-axis cannot be easily calibrated, since it depends on the volume of the sample, but the remanent magnetization can be estimated as a fraction of the saturation magnetization for each loop.



TENSILE AND SHEAR STRENGTH OF ADHESIVES

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Anne Arundel Community College
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PREREQUISITES

This experiment is conducted in a freshman-level course: Introduction to Engineering Materials. There are no pre-requisites for the course although students should have some knowledge of basic algebra.

OBJECTIVES

1. To tension and shear test adhesives.
2. To determine the tensile and shear properties of adhesives.

EQUIPMENT AND SUPPLIES

Tension testing machine.
Flat plate jaws for testing adhesive shear specimens.
Rod jaws for testing adhesive tension specimens.
Adhesive shear specimens.
Adhesive tension specimens.
Vernier calipers.

PROCEDURE

1. Measure dimensions of adhesive contact area for tension and shear specimens (mm).
2. Calculate contact area of tension and shear specimens and record results (mm²).
3. Install flat plate jaws in tension testing machine.
4. Mount adhesive shear specimen in flat plate jaws.
5. Slowly increase load on specimen until adhesive fails in shear. Note and record ultimate load (N).
6. Repeat steps 4 and 5 for the remaining adhesive shear specimens.
7. Install the rod jaws in tension testing machine.
8. Mount adhesive tension specimen in rod jaws.
9. Slowly increase load on specimen until adhesive fails in tension. Note and record ultimate load (N).
10. Repeat steps 8 and 9 for the remaining adhesive tension specimens.

RESULTS

1. Calculate ultimate tension and shear stress for all specimens (Pa).
2. Tabulate adhesive type, load type and ultimate stress.
3. Establish some conclusions about the effectiveness of the various adhesives tested relative to the material bonded.
4. Establish some conclusions about the load type (tension or shear) producing the best adhesive performance.

NOTES TO INSTRUCTORS

This experiment is best conducted on a tension testing machine with mounting jaws available for testing flat plate and rod specimens. Figure 1 shows the adhesive tension specimen. Figure 2 shows the shear specimens. Both are made from readily available aluminum. Dimensions of the specimens can be adjusted to the availability of material and load capacity of the tension testing machine.

Using a variety of adhesives will make the experiment more interesting. Purchasing types used in the home and in construction is easy. Obtaining types used for industrial manufacturing is more difficult. Lead time for bonding together tension and shear specimens should be long enough for adhesives to develop their full strength. Try to have similar surface finishes and clamping pressures for all specimens.

After the specimens are tested, many can be reused by removing adhesive and establishing a new substrate. This can be accomplished by wire brushing, surface grinding or sanding.

This experiment investigates the effect of load type and adhesive on ultimate strength. Other test variables can be introduced such as substrate type (wood and plastic), surface finish, cure variables and environmental conditions.

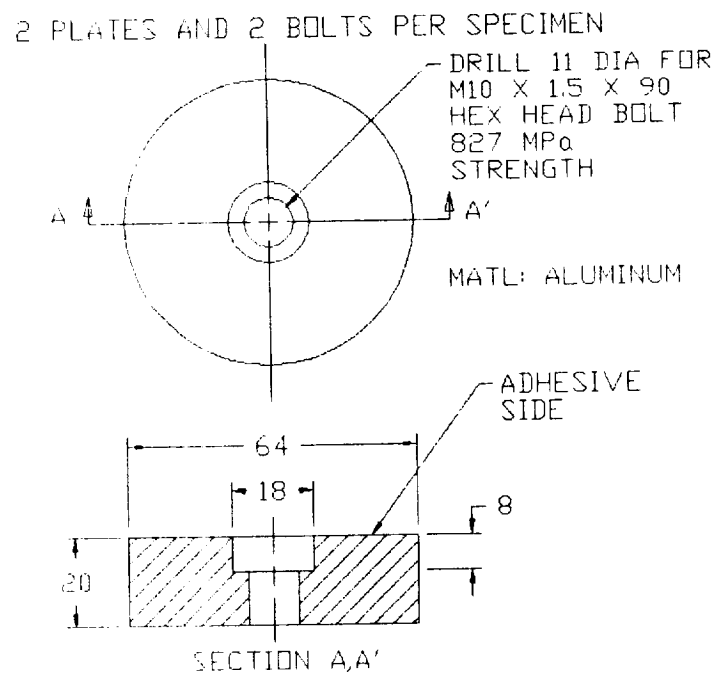
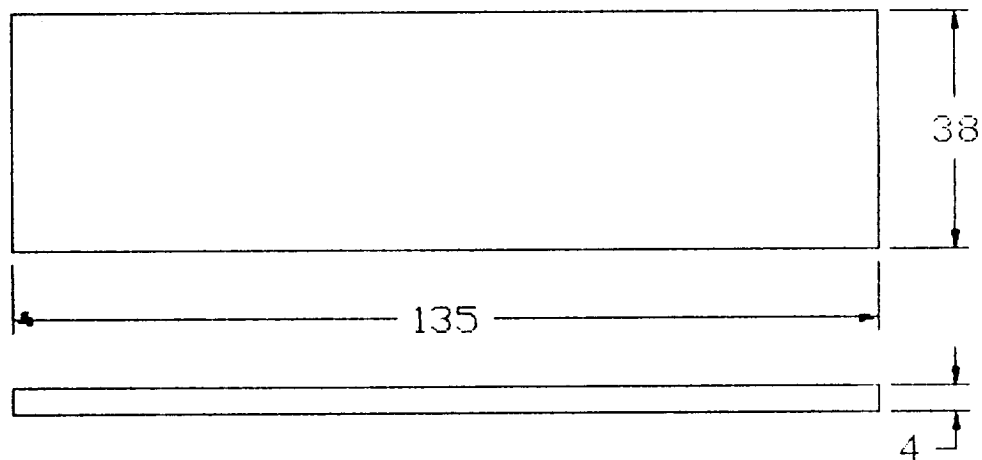


FIGURE 1
Adhesive Tension Specimen



MATERIAL: ALUMINUM
2 PLATES PER SPECIMEN
OVERLAP PLATES 38 mm FOR SPECIMEN

FIGURE 2
Shear Specimen

Experiments and Other Methods for Developing Expertise
with Design of Experiments in a Classroom Setting

John W. Patterson
Iowa State University
Ames, Iowa

INTRODUCTION

The only way to gain genuine expertise in "SPC" (Statistical Process Control) and "DOX" (the design of experiments) is with repeated practice, but not on canned problems with "dead" data sets. Rather, one must negotiate a wide variety of problems each with its own peculiarities and its own constantly changing data. The problems should not be of the type for which there is a single, well-defined answer that can be looked up in a fraternity file or in some text. The problems should match as closely as possible the open-ended types for which there is always an abundance of uncertainty. These are the only kinds that arise in real research, whether that be basic research in academe or engineering research in industry.

To gain this kind of experience, either as a professional consultant or as an industrial employee, takes years. Vast amounts of money, not to mention careers, must be put at risk. The purpose here is to outline some realistic simulation-type lab exercises that are so simple and inexpensive to run that the students can repeat them as often as desired at virtually no cost. Simulations also allow the instructor to design problems whose outcomes are as noisy as desired but still predictable within limits. Also the instructor and the students can learn a great deal more from the postmortem conducted after the exercise is completed. One never knows for sure what the "true data" should have been when dealing only with "real life" experiments. To add a bit more realism to the exercises it is sometimes desirable to make the students pay for each experimental result from a make-believe budget allocation for the problem.

Of course, the students find all this open-endedness and uncertainty very unsettling, but this is what most characterizes the kinds of investigations they will encounter later and it is important that these features be part of the students' lab experience. Better that the students' first encounters with these kinds of anxieties come in a college classroom under the tutelage of an experienced educator, than on their first job under the direction of a stressed out supervisor.

MECHANICAL SIMULATION

To be most effective, a lab exercise should be mechanical in nature and completely open to visual inspection while it is working. The reason is simple: All our experience is at the macroscopic level where mechanical phenomena dominate and it is largely by "seeing" that we have gained our experience. Hence, we take

data points more seriously when we can plainly see them being generated in a simple, easily understood fashion. Only later should one go to more exotic data production schemes, such as analog circuits or computers whose inner workings are usually much further removed from the world we experience.

One of the most effective teaching devices for introducing students to statistical methods is the so-called Quincunx, originally introduced by Sir Francis Galton back in the 1800's (see Figure 1). These simple machines allow one to generate endless sequences of samples, each drawn from a well defined population of known mean and standard deviation. Even easier to understand are the sampling boxes and sampling bowl schemes that are also in wide use today (see Figures 2-4). None of these are difficult to construct, but all are commercially available. For example, the Lightning Calculator Company of Troy, Michigan ([313]-649-4462) sells ready-to-use versions of the Quincunx, chip boxes, sampling boxes, and sampling bowls. They come completely assembled and are shipped with brief instructional brochures, an audio cassette, and ready-to-copy data forms for use in the lab. Statco Products, also of Troy, Michigan ([313]-879-7224) offers a similar array of such items.

Xbar-R charts for in-control processes are easily constructed using these devices and the students benefit from seeing the histograms take shape as the beads are dropped. Attributes charts (% defective, etc.) are best illustrated by sampling from a mixture of differently colored beads all housed in the same bowl. Specially constructed paddles make it easy to draw samples of various sizes. Sampling bowls can be used to construct and study p charts, u charts and c charts.

To control the attribute's mean frequency (% defective etc.), one simply adjusts the fraction of beads having a particular color in the sampling bowl. To examine the effects of different sample sizes one simply uses a different sampling paddle. With the quincunx, one controls the location of the bead drop by sliding it left or right. Different pegboards can be inserted to change the standard deviation of the Quincunx and the user decides on the number of beads dropped per sample. Again, the ease of operation of sampling bowls and the Quincunx makes it possible for the students to conduct as many runs as they care to and the data generation procedure is not nearly the time consuming distraction it is when actual experiments are used. Sometimes students dream up personal mini-research investigations to check various aspects of the recommended SPC and DOX procedures. Consider, for example, a scatter diagram study of the relation between the ranges and standard deviation of samples drawn from a Quincunx. Will the relation depend on the number of items per sample? Could this kind of information be used to convert R bar values into control limits on an Xbar-R chart? The possibilities are almost endless.

Also available from the Lightning Calculator people is a process simulation training kit called "prosim" (See Figure 5). It explains how the Quincunx can be used to demonstrate such statistical design strategies as One Way ANOVA, 2^2 and 2^3 factorial designs, two factor experiments with interactions, and Taguchi's L_8 and L_9 Orthogonal Arrays. After a little thought, however, it is easy to see how these instructions could be modified so as to simulate data for almost any kind of statistical design strategy in use today, including the so-called response surface methods.

COMPUTER SIMULATION

Once you see how the mechanical simulators work, it is a fairly simple matter to write programs that do the same thing either on a PC or on a hand held programmable calculator. Two software packages, a statistics simulator and an SPC simulator, were described in pp 84-5 of the July 1988 Quality Progress. In essence, they are computer versions of the Quincunx and run on IBM/PC/XT/AT compatibles having 128K of memory and one or more disk drives.

In my view, computer simulation methods are most useful for teaching and learning the strategies for designing multi-factor experiments and then interpreting the results. Many people insist that only real life experiments carried out with actual laboratory equipment should be used when teaching the design of experiments; however, I disagree. Restricting oneself only to real life experiments of the simplest sort, there is no definitive way to critically check the inferences one is led to with the DOX strategies he or she is employing. And if one tries to solve this problem by going to a very simple experiment--such as studying the period of a simple pendulum as a function of the mass, length and starting angle of the pendulum--the students simply go to the closest physics text to see what dependences are predicted from theory. This eliminates the most important aspect of the exercise, namely the haunting feeling one has about investigating some response variable without knowing whether--much less how--it depends on the controls being studied. Knowing these things beforehand completely changes the mindset of the investigator and can severely undermine his or her ability to proceed objectively.

By using tailor-made simulation programs, the instructor can generate noisy data from a well understood law such as the gas law, $V = nRT/P$. But the law should then be disguised by replacing V , n , T and P with Y , u , v and w respectively. Also, by adding a superfluous control variable or two, say x and z , (that do not appear in the gas law formula), the instructor knows to look for these as being totally insignificant factors in the experiment. If they do register significant effects, either the student or the DOX software must be doing something wrong. Afterwards, the students can be told that Y was really the volume of a perfect gas, u was the number of moles, v the absolute temperature, w the pressure and that x and z were dummy variables all along. When all this is revealed afterwards, the students find "the scales falling from their eyes", so to speak, and can then review the decisions they made in a totally different light, namely that of informed hindsight.

Here in undisguised form is a DOX problem based on the gas law. Shown in Figure 7 is the computer code I used to simulate noisy V data on a Casio fx-8000G (or fx-7000 G) hand held programmable calculator.

HANDS-ON, IN CLASS EXERCISE USING SIMULATED DATA

Design, execute and interpret an experiment to study the alleged dependence of gas volume ($V:10\ 350$)* on mole number ($n:0.5\ 2.0$), temperature ($T:300\ 1000$),

*The values over which the variables are to be ranged are bracketed by the numerical values following the colon. The same is true for the control variables n , T and P .

pressure (P:0.5 2.0), and xylene content (x:0.01.0). Assume we require a resolution of 1.5 units for the response variable V and that its estimated standard deviation is about 0.7.

- Steps:
- 1) Execute the resolution preliminaries.
 - 2) Design an experiment using a linear, factorial, quadratic or cubic design for all four of the control variables listed above (n,T,P,x). Use five replications.
 - 3) Conduct experiments at the control settings given your experimental design and analyze the data.
 - 4) Produce an effects table and analyze it to see if any of the four control factors has little or no effect.
 - 5) Produce contour maps of P vs T for n = 0.5 and 1.25 and shade in the P-T domain(s) for which V is less than 50 but greater than 30.
 - 6) Now use the ideal gas law to sketch in the "true" contours for V = 50 (or any other choice that meets your fancy) and compare these to the same contours on the response surface map.

In the MSE 341 course at Iowa State, we use a software package called EChip to teach expertise in experimental design. This will be described further below. The following problem is taken directly from the EChip text and the software contains a simulator program that can generate response data for whatever control variable settings the user wishes to specify. I include here the entire problem description but not the simulator program nor the extended text discussion of the problem. However, I think the reader will appreciate the realistic nature of the problem, which is based on an industrial problem that was successfully solved by an EChip user.

ECHIP PROBLEM 5: EXTRUSION OF A NEW THERMOPLASTIC

This is a wrap-up problem that ties together all you have learned so far. It should take about four hours to complete and everyone should be able to obtain a defensible solution using the methods discussed thus far.

Background

You work in R and D in the plastics division and have been asked to fully define the process for making a new plastic in one of the extruders on the compounding line. An extruder is like a giant meat grinder with a massive internal screw (auger) in the extended barrel. Raw materials are fed into the receiving funnel at one end; these are melted and mixed by a combination of heat added by elements on the barrel wall and by the shearing action of the auger. Everything eventually gets pumped out the extruder die (hole) at the exit end of the barrel.

The Control Variables

On talking with the inventor of the new plastic, you find he has worked only with small, lab scale equipment and that he has not defined the precise viscosity of the base polymer of the new plastic. Consequently, you will have to determine the percentage of the two additives add 1 and add 2. He also says that the moisture content of the base polymer (which you can control) often seems to be important.

You can control the temperature in each of four zones along the extruder barrel (T_1, T_2, T_3 and T_4) by automatic controllers on the heater elements.

Adding all this to information obtained from the operators of the extruder, you find there are at least ten control variables that may or not be important. Here they are along with the ranges over which they can be adjusted.

Add 1....	0.0% to 0.5%.	This is supposed to induce uniform melting.
Add 2...	2.0% to 4.0%.	This is a filler that is said to improve strength.
Viscosity...	The viscosity can be set anywhere from 60 to 80.	
Moisture...	The moisture content can be set from 0.1% to 0.25%.	
T_1 through T_4 ...	Each can be controlled at 260C min to 320C max, independently.	
Rate...	Feed rate of input is from 100 to 200 pounds/hr.	
RPM (auger)...	Can be set anywhere from 150 to 300 rpm.	

The Response Variables

On talking to your marketing people, you conclude that tensile strength (TS) is the most important property but measuring it requires a destructive test. It would be nice if the TS correlated with some easily monitored manufacturing variables so that the TS for each batch could be estimated from the other measurable variable. Old hands tell you they can pretty well estimate the TS of thermoplastics from the temperature of the melt during production. This is almost too good to be true so you decide to verify their claim by studying both the TS and the melt T as response variables. So your responses are chosen as follows.

Melt T...	Estimated range is from 280 to 325C
TS...	Ranges from 15000 psi to 30,000 psi with the minimum advertised specification being 25000 psi

Standard Deviation and Resolution

Discussions with marketing have resulted in a desired resolution of 10%. The general argument is that products less than 22,500 psi will be totally unacceptable. Since this is 10% less than 25,000 psi, the resolution of the experiment must be capable of detecting at least this difference.

You are fortunate in having a data base of tensile testing data from which you have calculated a standard deviation estimate of 0.02 for log 10 tensile strength. The antilog of 0.02 is approximately 1.05 which means that the standard deviation is about half of the desired resolution.

You may use 0.04 (the common log of (1.10)) for resolution and 0.02 for standard deviation in the number of trials calculations.

Final Hints

Make sure you have the goals clear. Many students, proceeding in haste, think optimization is the only goal. If you think this, reread p 5.4 very carefully.

Check it out. Try your model out and see how well it predicts.

All of the indicators may be positive. There may be no lack of fit indicated in the Effects Table, and the contour plots may make sense, but the model may still be in error. There is little redundancy in the recommended designs and they can fit the particular set of data but not the response surface from which the data comes; recording errors are a common problem.

The comments about the analysis of linear designs in the last paragraph of section 10.2 are important.

This problem should be worked several times. The first time without using design augmentation. Get fresh data at each stage. Once you have "solved" it this way and read Appendix B, you can redo it with design augmentation. For a third trial, you might try blocking-- once without blocking is recommended.

ECHIP'S DOX SOFTWARE

Many factors have conspired to keep undergraduate engineers from gaining knowledge of the DOX strategies they so badly need. For example, few if any engineering students have had the time or inclination to fulfill the prerequisites required for the DOX courses traditionally offered in statistics departments. This has tended to keep them out of formal DOX courses in college. Those engineers who have learned these methods have done so either as part of a graduate program or, more likely, by attending intensive short courses after graduation and at some employer's expense. Such short courses are marketed by numerous companies, consultants and continuing education units around the country. Interestingly, they learned to use DOX without taking all the traditional prerequisites. Also, more and more software packages with DOX capabilities are becoming available to the users of personal computers and these, too, can be used quite effectively by students who have not taken extensive prerequisite courses in statistics. In other words, engineers are bypassing departments of statistics and are learning DOX strategies on the job, either from short courses or through the use of software packages or both.

In an effort to better prepare our MSE students for what they will face upon graduation, we have purchased a license to use the EChip software package in our MSE 341 course. We feel its statistical methods are more than rigorous enough for our purposes and we especially appreciate EChip's emphasis on strategies rather than statistics or calculation procedures. The software focuses more on how to make the key decisions when designing and executing experiments and leaves the computational details and statistical analyses to the computer and keeps them "behind the scenes", as it were. After first deciding whether the proposed investigation will fit within the available budget, the user is directed on a swift but systematic course of action through the cycle shown in Fig. 8. An extensive array of response surfaces produced in the form of contour maps that can be scrolled, sorted through and

interacted with in the most useful fashion. Upon completion of the study, one decides on whether or not another battery of experiments should be undertaken. If so, the procedure is repeated, though in a modified form that accounts for what has been learned or what variables may be left out.

EChip provides a choice of several standard designs plus a powerful algorithmic design option for use when none of the standard ones can possibly suffice. This can occur when certain regions of the control variable space simply cannot be sampled due to unavoidable technical difficulties. EChip enables the user to disallow whole groups of troublesome control variable settings and then to algorithmically devise an optimal design in the subregion of settings that is left. Algorithmic designs are also used when mixture variables are to be included or when one wishes to augment the design executed in the previous iteration.

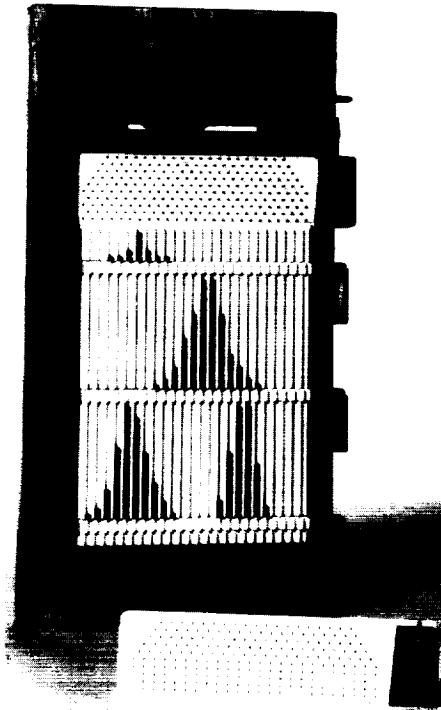
As EChip is response surface oriented, the response variables (several may be studied simultaneously) must be of the continuous type. Some but not all the controls may be of the categorical variety but the software is intended for studies involving continuous control variables for the most part. EChip's ability to handle both mixture and nonmixture controls simultaneously makes it especially useful for materials and chemical engineers.

CONCLUDING REMARKS

In the foregoing sections, I have promoted the use of simulation schemes, first of the mechanical-visual sorts and later of the computer-numerical types. In my view, these are the best ways to generate response data for multifactor experiments. Since the instructor specifies everything (including the noise level!) in the model that generates the data, it is relatively easy to diagnose the mistakes that arise in the students' work because there is no possibility for poor lab methods or experimental equipment failures to cause problems. Discrepancies can only be due either to software bugs or to inadequacies in the DOX strategies being used or to mistakes or poor judgment by the user. I, for one, very much favor the simulation approach when trying to help students acquire expertise in the use of DOX methods for analyzing or optimizing complicated multifactor processes. On the other hand there is indeed a lot to be said for also including real experiments in such a course, despite all the difficulties they may entail. For this reason, I conclude by citing a number of candidate experiments we are hoping to incorporate in the MSE 341 course in future offerings. Unlike the examples discussed so far, these place much greater emphasis on mixture variables.

- 1) Study the dependence of AC conductance and capacitance for aqueous solutions ($\text{H}_2\text{O} + \text{NaCl} + \text{sugar} + \text{H}_2\text{CO}_3 + \text{etc.}$) on temperature ($^{\circ}\text{K}$) compositions (mixture variables), AC frequency (H_2), geometric variables (distance between electrodes, their areas, etc.).
- 2) Study AC conductance, capacitance and density of moist soils ($\text{H}_2\text{O} + \text{sand}$, silt, etc.) and their dependences on compaction pressure, water contact, soil type, AC frequency, etc.
- 3) Study dependence of freezing point depression of aqueous (or other) solutions on composition variables.

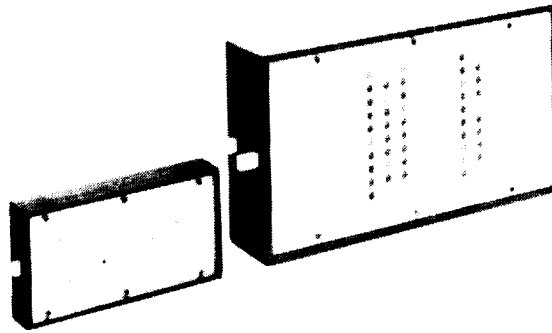
- 4) Study dependence of density and permeability of sintered TiO_2 (or ZnO , NaCl , etc.) powders on binder type, compaction pressure sintering temperature, sintering time, particle size distribution, etc.
- 5) Study dependence of final density and porosity of hot pressed pellets of bakelite powder on hot pressing temperature, pressure and time.



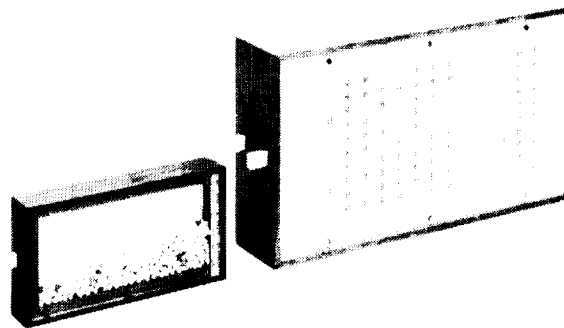
THE QUINCUNX

Figure 1. The Quincunx.

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Front



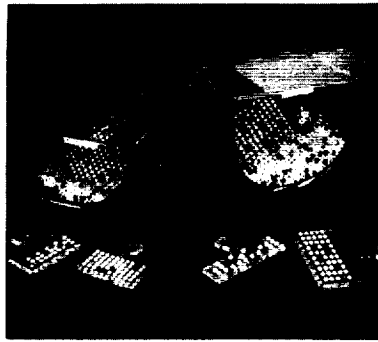
Back

THE SAMPLING BOX

Figure 2. The sampling box.

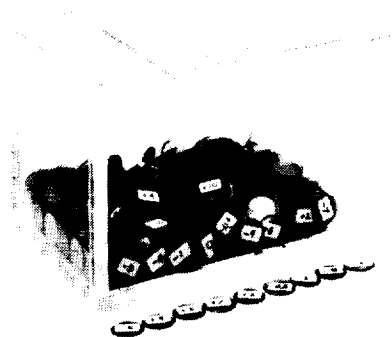
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THE SAMPLING BOWL

Figure 3. The sampling bowl.



THE CHIP BOX

Figure 4. The chip box.

PROSIM, which stands for process simulator, is a training aid that uses a quincunx to teach designed experiments.

The PROSIM kit includes :

- Master Forms to be used by the instructor and student.
- Overhead Masters for instruction and demonstrations.
- A "D" ring binder for the 140 plus page manual.

The training package includes step by step procedures for how to set up any quincunx with a variety of factors and degrees of significance. The handy setup forms and work sheets will help simplify any design experiment class. The data analysis forms included treat conventional designed experiments and Taguchi Orthogonal Arrays.

The image shows four overlapping worksheets from the PROSIM kit. From left to right, they are: 1. 'FUNNEL POSITION SETUP SHEET' with a grid for funnel positions. 2. 'L8 EXPERIMENTAL DESIGN WORKSHEET' with a grid for L8 orthogonal array experiments. 3. 'L4 & L8 EXPERIMENTAL DESIGN WORKSHEET' with a grid for L4 and L8 orthogonal array experiments. 4. 'OWA EXPERIMENTAL DESIGN WORKSHEET' with a grid for One-Way ANOVA experiments. Each worksheet has various sections for data entry, calculations, and analysis.

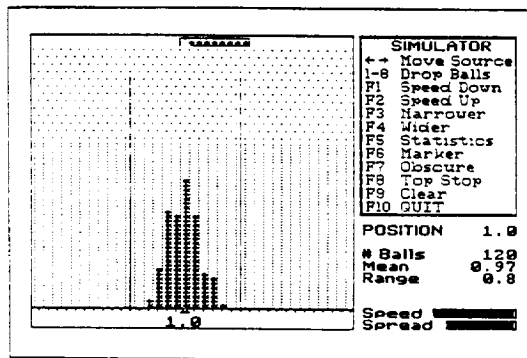
The detailed procedures included with PROSIM address the following topics:

1. How to set up and scale the quincunx.
2. One way ANOVA.
3. 2^2 Full Factorial Designs.
4. 2^3 Full Factorial Designs.
5. Two factor experiments with interactions.
6. Taguchi L_8 Orthogonal Arrays.
7. Taguchi L_9 Orthogonal Arrays.

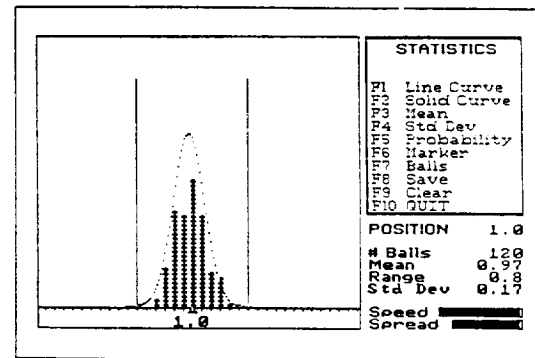
PROSIM

Figure 5. Prosim.

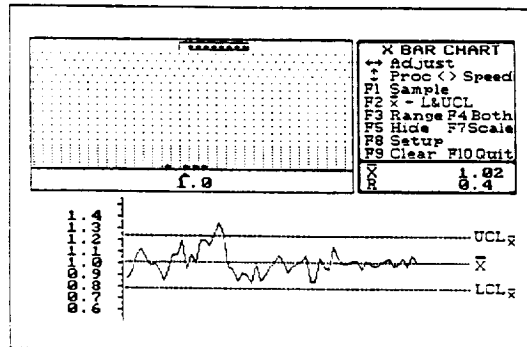
Two Quincunxes for the PC



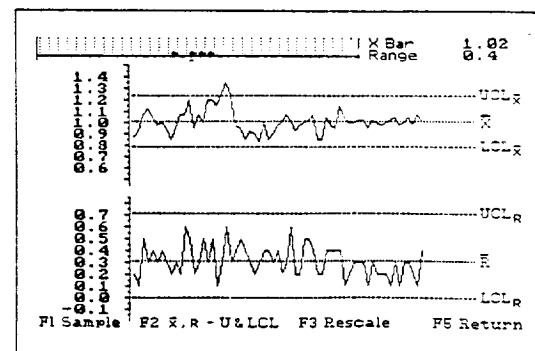
This is the bead frame in the Statistics Simulator. It shows a sample of 120 centered on 1. The standard deviation can be varied by using the F3 and F4 keys. Upper and lower spec limits were drawn in using the F6 key.



This is the Statistics Simulator analysis screen based on the information in Figure 1. Plus and minus standard deviation lines also can be drawn in, and the proportion between any two limits can be calculated.



The Statistical Process Control Simulator produces an X-bar chart with limits that can be updated. The effect of adjusting the mean, spread, or both can be demonstrated easily.



Pressing the F5 key displays the X-bar and range charts. Menu selections are always visible on the screen in the Statistical Process Control Simulator.

Figure 6. Two Quincunxes for the PC

Figure 6 reprinted with permission, *Quality Progress*, July 1988

PROG 5	Comments and Remarks
Mc1 "SD"?→S Lb1 1 PROG 3	Clears all memory registers Prompts for STD. DEV. "(TRUE)" and stores it in S PROG 3 is a subroutine that returns a number "A" drawn from a normal (0.1) population
"n"?→N "T"?→T "P"?→P "x"?→x	Prompts for a value of n and stores it in reg. N " " " " " T " " " " " T " " " " " P " " " " " P " " " " " x " " " " " x
.082NT ÷ P→V V + S × A→V	Calculates the "TRUE" value of V and stores it in V Adds a normal residual noise contribution (STD DEV = S) onto V(TRUE) and stores <u>that</u> in V
"V=" :V Δ GOTO 1 (CASIO fx-7000G)	Displays V = and the value just simulated cycles back to Lb1 1 for the next simulated experimental result
PROG 3	Comments and Remarks
ϕ→A:48→F:Lb12 A + RAN#→A DS2 F:GOTO 2 (A-24) ÷ 2→A (CASIO fx-7000G)	Sets A to ϕ and counter, F, to 48 Adds RAN# (uniform in int. 0-1) to register A Decreases counter By 1.0 when F > ϕ, GOTO Lb12 When F drops to ϕ, convert the sum in A to a normal (0,1) value and store it in A for use in the main program.

Figure 7. A program with subroutine for simulating volume data with a known variability. Assumes $V = \pi RT/P$.

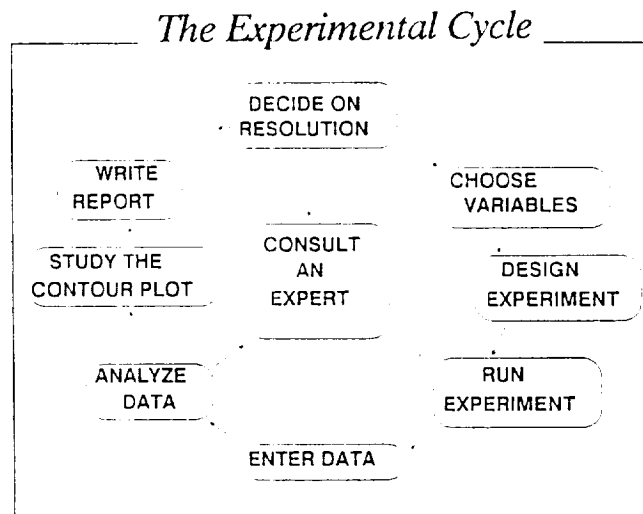


Figure 8.

MANUAL AND COMPUTER-AIDED MATERIALS SELECTION
FOR INDUSTRIAL PRODUCTION:
AN EXERCISE IN DECISION MAKING

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San Jose State University

Summary

Students are introduced to methods and concepts for systematic selection and evaluation of materials which are to be used to manufacture specific products in industry. For this laboratory exercise, students are asked to work in groups to identify and describe a product, then to proceed through the process to select a list of three candidates to make the item from. The exercise draws on knowledge of mechanical, physical, and chemical properties, common materials test techniques, and resource management skills in finding and assessing property data. A very important part of the exercise is the students' introduction to decision making algorithms, and learning how to apply them to a complex decision making process.

Significance

Materials selection is the process of choosing materials from which to make products in industrial production. A familiarity with materials selection is critical to product designers, to product and manufacturing engineers, and to industrial technologists who will have close review over production lines and problems. In addition, a study of the decision making involved is sound basic education for today's college students.

The Problem

Ten years ago, when this researcher first started a study of the materials selection process, very little in the way of computer tools was available to aid the designer or engineer in the process. At that time, an article in Metals Progress¹ described an early but well-designed system for polymer selection used at John Deere's corporate headquarters in Moline, Illinois. Other true systems for selection were manual in basis, including several card-based methods. The most notable of these was developed by H. Laurie Miller of Northern Telecom, in Canada². Mr. Miller explored several manual methods, including transparent overlays to identify matches to requirements and slotted cards for appropriate materials. None of these methods were adequate to deal with the large quantities of data involved: They are even less adequate today.

Manual Methods

In spite of the problems, most industries continued, as they do today, to select materials for production of products without the use of computing tools. There are essentially only four 'practical' approaches to materials selection in use throughout industry:

1. Use the material that was used last time
2. Use something that is in stock or heavily used for other products
3. Call in a materials supplier to evaluate the problem and recommend a suitable material.
4. Use an in-house expert or hire a consultant who has training in materials selection (the materials 'expert').

The consequences of the first three approaches will be clear upon a moments introspection. Formal university training in materials selection (option 4) is strangely rare. The program in Industrial Materials at the Division of Technology in San Jose State University attempts to give each graduating student an introduction to the rudiments of manual methods for materials selection. Because this field can so easily be expanded to encompass stress analysis, mechanical design, and other disciplines, it is important to distinguish the method of selection from the methods for product design. Our students are given the handout that follows this report to help them learn and execute the selection process.

Computer Solutions

In 1978, the first Apple II microcomputers were beginning to be marketed. No one even dreamed that IBM Corporation would ever show any interest in the "toy box" computer market. In that context, the only computer systems powerful enough to handle the computation, and especially the data storage and manipulation required for materials selection, were large minicomputers and mainframes. Such large systems were controlled by the data processing staff. For this reason, early studies showed very limited user access to such systems even where they existed, and the cost of system development was exorbitant for most engineering and design groups.

In 1981, the researcher established that it was indeed possible for a computer program, given appropriate input, to select materials from a database which will perform competitively to materials selected manually by professional materials engineers. In other words, given the parameters of the study³, the computer could successfully select materials very similarly to expert materials applications engineers. The algorithm developed for use in the program for this research was refined from several studies from all over the country and even in England. It also incorporated studies of the decision-making methods used by a number of expert materials selection engineers from John Deere, Amana, and Rockwell Collins Avionics Division.

An algorithm is a mathematical model of a process, which in this case can be applied as a rule by the program as it evaluates data from a database. It constitutes a decisionmaking rule, and as such it represents an effort to establish some artificial, expert system capabilities for materials selection. In the research program, the computer will attempt to minimize 'Z' in the equation

$$Z = \sum_{i=1}^n |(X_i - Y_i) / Y_i|$$

as 'i' varies from 1 to 'n' properties. This equation is the main algorithm used by the program to evaluate materials. X_i is a material property found in the databank, and Y_i is provided by the selector as a target specification for each property during program startup. Minimizing the value of Z gives us the materials with properties that offer the smallest total percent deviation from specifications.

This rule is simple and easily executed by the computer, but it is not adequate by itself, as it assumes that each property is equally important in each selection application. Since this is not the case, we must add a weighting factor to the formula so that deviations become more or less significant depending on the importance the selector assigns to each property for a given application.

The resulting algorithm is effective, but to perform this numeric calculation on each property for each material in the database can consume huge amounts of computer time, delaying user response time unacceptably. The algorithm is thus augmented by a simple screening tool which eliminates from consideration for any given problem all materials which do not meet specific absolute property requirements stated by the selector (user). This allows us to quickly reduce the number of materials that need to be screened. The computer will not take as long to screen the remaining candidates. As a matter of practical concern, A Hewlett Packard model 2000 time sharing minicomputer, very primitive by today's standards, using the BASIC language, never required more than three minutes to evaluate up to ten property specifications for over 300 candidates (even when time sharing).

In today's microcomputer environment, however, any high-end desktop computer can be configured as a basis for cost-effective materials selection. Minimum system requirements will depend mainly on the size of the database used, but could easily be met by a Macintosh SE/30 or better, or by a PC AT running at 8 MHz or more, and with a rapid-access hard disk drive of 20 or more Megabytes capacity. Such computer systems can also support programs that have recently been released by the American Society for Metals (ASM)⁴, by Corth Publishing Group (the D.A.T.A. series)⁵, and by others who have attempted to develop commercial solutions.

Future Problems

Although the researcher is attempting to develop a PC-based system that will evaluate materials from all groups (metals, plastics, and others), this has not been accomplished in the commercial market. Many factors contribute to the difficulty, but the principal one is that for each group of materials, different test standards, test methods, and data presentation are used. It is easy to develop some conversion or translation tables, but others are nearly impossible. New test standards and procedures will be necessary before such a system can really work well, and this may never be workable given the significant differences that exist between the material families.

Nonetheless, workable PC-based models for materials selection from a limited database can be developed in any classroom and engineering group by a single programmer with a little development time. This will remain an exciting field for further study for several decades to come, due to the enormous benefits that methodical materials selection offers for enhancement of productivity and product liability stature.

Conclusions

Students can undertake real selection problems based on these laboratory techniques, and those with some programming proficiency can develop prototypical computer-based selection systems. The study of these methods is of use to product engineers, manufacturing methods engineers, product designers (including industrial designers), and manufacturing managers and can result in great cost savings and in a more secure liability position through improved documentation.

References

1. Unterweiser, P. M. (April, 1977), Computer aided engineering at John Deere: A materials selection data system. Metal Progress, pp. 38-43.
2. Miller, H. Laurie (February, 1975), Systematic selection of plastic resins. Canadian Plastics, pp. 20+.
3. Bates, S. P. (December, 1981), A feasibility study of a system for computer-aided selection of materials. Dissertation, University Microfilms, Ann Arbor, MI.
4. A. S. M. (1988), MetSel, A computer program for metals selection. ASM, Metals Park, OH.
5. Corth Publishing Group. D. A. T. A. , Tools for plastics selection.

Student Handout for
Materials Selection Problems
Dr. Seth Bates

I. Abstract

This paper is a guideline for the solution of materials selection problems. Materials selection, in this context, means choosing a material from which to make a part or product in industrial production. The material chosen, clearly, must be able to withstand reasonable stresses during the life of the product, and not fail catastrophically where life or health of users is endangered.

In order to conduct the selection, you will need to think through every aspect of the part. From this you will come up with a list of properties which the material should exhibit. Your technical report should explain what the criteria are, how you arrived at them, and how you went through the process of choosing the one best material for the application.

Materials Selection Problems

II. Introduction

The selection of a material for a specific application involves methodical, thorough, and imaginative thinking. It should be approached as a big challenge to your knowledge and abilities. There are many models (procedures) available for selecting materials. This document provides you with two tools to use:

- a. a model, or set of procedures, which you will use to arrive at good solutions
- b. guidelines for writing up your selection report.

III. A Model Procedure for Materials Selection

This model is presented in a number of steps which are identified as

- Part or product identification
- Properties specifications development
- Coarse screening of families
- Fine screening
- Final identification
- Reiterations

IV. Part or product identification. Identify the part or product to be manufactured. Try to use parts or products which involve only one material, for simplicity. Prepare a simple but clear sketch or drawing. Describe in detail:

- a. The functions of the part - what it is and what it does
- b. The operating environment - in terms of the mechanical, physical, and chemical forces that will act on the part
- c. Any accidental forces that might reasonably be expected, and any accidental forces that might not be expected, but could have serious consequences
- d. The potential users of the product. Indicated age range, education or training level, or any other significant factor

V. Materials specifications development. Using information from the last section (b. and c.), list in detail the types of properties and characteristics that the material must exhibit, and the values or ranges of values where possible. The following list will help:

<u>Properties</u>	<u>Characteristics</u>
a. mechanical	d. fabrication qualities
b. physical	e. economics and aesthetics
c. chemical	f. any other relevant data

For examples of relevant properties, consider those listed in the references found in the reference list. Clauser's book, other Materials Science texts, and the Materials Reference Issue are not adequate sources of information for these data.

VI. Coarse screening. Identify those properties which must be met (called 'go/no-go' properties). Using the go/no-go's, evaluate the major families of materials to determine which actually offer candidate materials from which to make your part. The families include:

- | | |
|-------------------------|------------------------------|
| a. metals - ferrous | e. thermoset polymers |
| b. metals - nonferrous | f. thermoplastic polymers |
| c. ceramics and glasses | g. polymer composites |
| d. elastomers | h. woods and wood composites |

Then, using the go/no-go's and the databanks (references) listed above, identify specific materials from the families which passed the coarse screening. Use correct names, not tradenames (e.g. acrylic or acrylonitrile, rather than Plexiglass). You may have as many as 15 to 30 candidates (materials) at this point. Try to be selective enough to keep the number small.

VII. Fine screening. Rate all the properties that are still significant (some may not be) in terms of relative importance. Using these relative ratings, evaluate the candidates. Use Clauser's system (1975, p. 19). Identify the best 3 to 5 candidates and rank them.

VIII. Final identification. Compare the three to five final candidates on the most important points. If they all seem equally good choices, consider more subtle aspects such as aesthetics and economics. Rank them according to their relative value in this application, and explore the pro's and con's of using each one.

IX. Reiterations. Every materials selection involves compromises. At any point in the coarse or fine screening process you may see that some specification has unnecessarily limited the choice of materials, material types, or families. If this has occurred in your case, reconsider the original part description:

- ~Could the part be designed differently, to allow looser (or different) specifications?
- ~Perhaps aesthetics could be satisfied by a different method of finishing.
- ~Has your selection in fact identified a suitable material?

If these or other considerations apply, consider a redesign or reevaluation (reiteration). You should be flexible, and willing to change your part design. The selection of materials is a dynamic process.

X. Reference List for Selection Handout

ASM, (annual) Metals Handbook, vol. 1, Properties and Selection. Metals Park, Ohio

Clauser, H. R. (1975) Industrial and Engineering Materials. New York: McGraw-Hill.

The following are also useful:

The Materials Selector Issue of Materials Engineering (annual), Penton, Cleveland, OH.

The Modern Plastics Encyclopedia (annual), McGraw-Hill, New York, NY.

The International Plastics Selector (annual), Cordura Publications, San Diego, CA.

End of Handout

SCANNING X MICROSCOPY

Slide and Video Presentation by F. Alan McDonald
IBM Research Center

Dr. McDonald presented a slide/tape presentation on the variety of scanning microscopy techniques and the research being conducted at the IBM Thomas J. Watson Research Center in Yorktown Heights, New York, and other research developments throughout the world in scanning X microscopes.



Please refer to Scanned-Probe Microscopes, Scientific American; October 1989, pages 98 through 105, for the formal presentation of the topic.

This article, by H. Kumar Wickramasinghe, Manager of Physical Measurements at the Yorktown Heights Center, explains how scanned-probe microscopes examine a surface at a very close range with a probe which may be just a single atom across. These microscopes can resolve features and properties on a scale beyond other microscopes.

PREPARING TECHNICIANS FOR
ENGINEERING MATERIALS TECHNOLOGY

James A. Jacobs
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Norfolk State University
Norfolk, Virginia

Carlton H. Metzloff
Erie Community College - North
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Materials science and engineering is definitely among the worlds hottest technologies; the other two being biotechnology and communication technology.

Every expert interviewed [deans of engineering schools, school placement directors for engineering and heads of firms that emphasize advanced technology and employ significant numbers of engineers] had materials at or near the top of the list [top 10 careers for 1990s]. The burgeoning need for new materials stretches from high performance, specialty applications to cheap, high-volume substances for mass production. The implications for use and adaptation are unlimited... focused on developing ceramics, metal alloys, polymers, biological substances and other crystalline and amorphous materials. Beyond creation of such breakthroughs, there will be plenty of jobs in bringing these materials up to mass production and introducing them into manufacturing, processing, power systems, construction and other areas of application. (Paul Price, Graduating Engineer, March, 1988.)

A long held principle is that for every engineer and scientist there is a need for ten technicians to maximize the efficiency of the technology team for meeting needs of industry and government. Developing an adequate supply of technicians to meet the requirements of the materials related industry will be a challenge and difficult to accomplish.

A variety of agencies such as the National Institute of Standards and Technology (NIST formerly NBS), NASA, ASM International and Battelle Laboratories feel the need and wish to support development of engineering materials technology programs. In a joint effort among Battelle Laboratories, Department of Energy (DOE) and Northwest College and University Association for Science (NORCUS), for development of an engineering materials technology program for vocational programs and community colleges for the Pacific Northwest Region was recently completed. This effort has implications for a national model.

Curriculum

The increasingly interdisciplinary character of research [in materials science and engineering] has created serious problems of communication. Physicists often do not fully understand what chemists mean by terms like oxidation potential, and chemists often do not understand physicists' calculations involving path integrals and thermal Green's functions. Physicists and chemists attach different meaning to the term resonance. Electrical engineers understand logic circuits and switching devices but may know little chemistry and nothing about ceramics. Ceramicists understand materials and processes, but may not grasp electronic circuits or device physics. A period of adjustment will be needed for the different constituencies to learn to work together more effectively. (John P. McKelvey. "Understanding Superconductivity", Invention & Technology. Spring/Summer. 1989. p.56.)

The communication problem stated here involves MSE. Engineering materials technology at an associate degree level of education is even newer. A curriculum for technicians could take many forms in an attempt to meet the requirements of many areas related to materials science and engineering. Among the few programs, Erie Community College and Hocking Technical College are offering AAS degrees; the Texas State Technical Institute System is establishing a program at its Sweetwater campus.

The model Associate of Applied Science degree in Engineering Materials Technology shown on the attached sheet provides a general structure. It purposely has course titles which need delimiting while also including a core of courses necessary to develop cognitive, affective and psychomotor skills with the underlining principles of math, science and technology so students have job entry skills, and so that students can learn about and adapt to evolving technology.

Needs analyses, advisory committees and other similar input are required to delimit the program in order for it to serve specific needs. For example, two courses each are shown for materials processing and manufacturing processing. This leaves room for covering the hundreds of processes in materials technology. But community or regional needs would influence whether there was an emphasis on metal working, plastic processing or perhaps electronic materials such as semiconductors.

Many materials technologies are emerging. They will compete with established but evolving technologies which have their special interests to promote. For example, composites are being designed and promoted to replace metals. But the established metal industry is developing new alloys, more efficient processing techniques and means to keep them competitive.

Students

As mentioned above, due to a lack of public awareness about materials technology in general and of the career opportunities in MSE in particular, it may be difficult to attract students into a new program. There are on-going efforts such as the career outreach program by ASM International and the course Materials Science Technology in the Northwest Pacific that have been designed to increase awareness of MSE.

On the Virginia Peninsula a master technician program was developed with a 2 plus 2 plus 2 structure involving vocational schools, community colleges and universities. We developed two materials and processes technology curriculum guides for the Commonwealth of Virginia's Technology Education Service and have also written numerous modules on engineering materials technology which have been published in The Technology Teacher. These modules can be used to develop awareness in high school and introductory college levels of science and technology. There are other means to promote the career opportunities in materials technology including scholarships and contests that emphasize MSE.

ENGINEERING MATERIALS TECHNOLOGY
ASSOCIATE OF APPLIED SCIENCE DEGREE

FIRST YEAR

First Semester

Communication	3
Mathematics	3
Chemistry	4
Engineering Materials Technology	3
Physics	<u>4</u>
	17

Second Semester

Communication - Technical Reporting	3
Mathematics	3
Chemistry	4
Processing of Materials	3
Computing in Engineering Technology	2
Static & Strength of Materials	<u>3</u>
	18

Summer Session

Cooperative Education	3
-----------------------	---

Second Year

First Semester

Manufacturing Processes I	4
Non-Destructive Evaluation	3
Social Science	3
Metals and Polymers	4
Materials Handling, Safety & Environment	<u>4</u>
	18

Second Semester

Materials Processes II	4
Ceramics and Composites	4
Manufacturing Processes II	3
Engineering Materials Technology Project	3
Elective or General Education	<u>3</u>
	17

TOPICAL OUTLINE

Preparing Technicians for Engineering Materials Technology

I. History of Materials Science Department

- A. 1947 - Erie County Technical Institute
1960's - Changed to Erie Community College
- B. 1947 - Department of Metallurgical Technology
1960's - Changed to Materials Science Technology

II. Curriculum Requirements

Math	- Technical Mathematics I Intermediate algebra & trigonometry.	4.0 credits
Math	- Technical Mathematics II Intermediate algebra & trigonometry.	4.0 credits
Physics	- Technical Physics I Friction, fluids, sound thermo- dynamics, heat transfer.	4.0 credits
Physics Lab	- Experiments in Mechanics Heat & sound.	0 credits
English	- College Composition	3.0 credits
English	- Composition & Reading in Science & Lit	3.0 credits
Chemistry	- Introductory College Chemistry I For students that require working knowledge of chemistry. Includes: chemical bonding, chemical equations, stoichiometry & gas laws.	3.0 credits
Chemistry	- Chemistry I Laboratory Topics covered: measurements, physical properties, specific heat, atomic weights, chemical reactions, etc.	1.5 credits
Chemistry	- Introductory College Chemistry II Topics covered: solutions, acidi- metry & alkalimetry, oxidation- reduction, chemical equilibrium, organic chemistry, metal & polymers.	3.0 credits

Chemistry - Chemistry II Laboratory Covers: solutions, acid & base titration, intro to organic compounds & qualitative analysis of transition metals.	1.5 credits
Social Science - Macro & Microeconomics - General Psychology	3.0 credits
Materials Science I - Intro to Materials Science The basic study of structures of materials and how these relate to their properties.	3.0 credits
Materials Science II Indepth study of all materials.	5.0 credits
Materials Science II Laboratory Metallurgical sample preparations and microscopic study of metallurgical specimens.	1.5 credits
Advanced Processes & Materials Materials joining covered in depth, as example welding and adhesive bonding. Nonmetallics as polymers, elastomers, ceramics & composites. SPC, product liability and product reliability are covered.	2.0 credits
Advanced Processes & Materials Laboratory Use metallographs & MP ₄ camera used to study polymers, ceramics & composites. Join metals with several welding processes.	2.0 credits
Strength of Materials Study of various mechanical properties of common materials. Stress-strain relationships, yield strength, elastic limit, modulus of elasticity.	3.0 credits
Strength of Materials Laboratory Formal report writing on tension, compression, shear, hardness, torsion, bonding impact & fatigue experiments. Use of equipment is stressed.	1.0 credit
Microprocessor Applications in Materials Science It is based on the premise that computers are a useful tool for solving, evaluating & modeling problems in Materials Science Technology.	3.0 credits
Non-Destructive Inspection Liquid penetrant, ultrasonic, eddie current, magnetic particle, x-ray and gamma radiation techniques are studied.	3.0 credits

Non-Destructive Inspection Laboratory Application & operation of equipment, interpretation of results included in laboratory procedures.	1.0 credit
Metal Casting A course in elementary metal casting practices covering pattern design, molding processes, sand testing, gating & risering & solidification.	1.0 credit
Metal Casting Laboratory Many non-ferrous alloys are poured into green sand mold, ceramic molds, metal molds & graphite molds. Electric arc, induction & gas crucible furnaces are used.	2.0 credits
Casting Processes All the industries that are associated with the manufacturing of castings are studied. This would include refractories, ceramic, plastics, rubber, wood & cement industries.	1.0 credit
Casting Processes Laboratory Steel, gray iron, ductile iron & stainless steel produced & made into useful objects. The senior research project will test the resources, intelligence, ingenuity & ability of students to work as a team.	2.0 credits
Machine Tools & Drawing Fundamentals of mechanical drawing with emphasis on blueprint reading. Intro to basic machine tools, lathe shaper, mill, grinder & drill press.	1.0 credit
Metallurgical Instrumentation Indepth study of strain gages, carbon analysis, micro-hardness testing, pyrometry, creep & emission spectroscopy.	3.0 credits
Metallurgical Instrumentation Laboratory Perform tests on installation, calibration & operation of instruments.	1.0 credit
Industrial Metallurgy Visitations to materials science plants.	1.5 credits
Manufacturing Processes I Introductory course covering the fundamental manufacturing methods, processes & equipment.	1.0 credit
Manufacturing Processes I Laboratory The laboratory will afford the students hands-on experience with forging, casting, powder metallurgy, etc.	1.0 credit

- Manufacturing Processes & Materials II 1.0 credit
An advanced course covering automated systems manufacturing, example - flexible manufacturing & computer-integrated manufacturing.
- Manufacturing Processes & Materials II Laboratory 1.0 credit
Topics in control systems, pick & place operations, automated assembly & computer-integrated manufacturing.
- A Survey in Computer-Aid Design 1.0 credit
A five-week survey of basic CAD tasks necessary to complete simple orthographic projections using micro CAD systems.

FUTURE AUTOMOTIVE MATERIALS - EVOLUTION OR REVOLUTION

P. Beardmore
Research Staff, Ford Motor Company

Abstract

An exciting era is evolving in the application of new materials technologies to automotive applications. The desire on the part of the automobile industry to completely satisfy the customers while concurrently meeting increasing demands and regulations for stringent emission control and fuel efficiency is opening a plethora of opportunities for new materials. In many cases, materials solutions are the only mechanisms for resolving some of the upcoming issues. The materials scientist and engineer will therefore have a primary role to play and will assume a position of significance hitherto unseen in the automobile industry. The nature of the industry dictates that changes are primarily evolutionary with respect to chronology but nevertheless some of the future material changes will be revolutionary in nature. This presentation will treat three primary systems of the vehicle separately, based on the different materials approaches which will be adopted. These areas are (1) skin panels, (2) body structures and (3) powertrains. The competition between a variety of new materials in these 3 systems will be discussed in detail with the various tradeoffs being outlined. Amongst the more prominent of the new breed of materials will be new steel technologies, structural plastics (FRP), aluminum alloys (conventional and rapidly solidified), titanium alloys, metal matrix composites and smart materials (electrorheological fluids, etc.). The pace of development and application is accelerating rapidly and the impetus is likely to increase. Exciting materials days lie ahead!

NINETIES INFLUENCES

- Total Customer Satisfaction
- Competition
- Environment
- CAFE

APPLICATION STRATEGIES

- Skin Panels
- Body Structures
- Engine Components

APPLICATION STRATEGIES

- Skin Panels

- Body Structures
- Engine Components

FUTURE AUTOBODY MATERIALS MAJOR ISSUES

- Cost Effectiveness
- Lightweight
- Environment/Recyclability

POTENTIAL AUTOBODY MATERIALS

- High Strength Steels
- Engineering Plastics
- Structural Composites (FRP)
- Aluminum Alloys

SKIN PANELS MAJOR ISSUES

- Cost Effectiveness
- Formability
- Functionality (Stiffness, CTE, Dent Resistance, Oil Canning)
- Lightweight
- Handleability/Damage Resistance
- Class A Surface Finish
- Paintability (Paint Adhesion, Etc.)
- Heat Resistance
- Repairability
- Recyclability

SKIN PANELS

- Mild Steel
- Bake Hardenable Steels
- SMC
- Thermoplastics
- Aluminum Alloys

FUNCTIONALITY

- Stiffness
 - Overall Panel/Closure Panel Stiffness
 - Local Panel Stiffness (Oil Canning)
- CTE
 - Fit and Finish
 - Interference
- Dent Resistance
 - Yield Strength Controlled

STEEL SKIN TECHNOLOGY

- **Materials**
 - Mild Steel
 - Bake-Hardenable Steels
- **Advantages**
 - Cost
 - Quality Control (e.g. Continuous Anneal Lines)
 - Formability
 - Experience (Vast Data Base)
 - Improved Dent Resistance (B-H)
- **Limitations**
 - Weight (?)
 - Deep Drawability (B-H)

PLASTIC SKIN PANELS

- **Materials**
 - SMC - Horizontal Panels
 - T/P - Vertical Panels
- **Advantages**
 - Design and Styling Versatility
 - Existing Experience Base
 - Few Structural Issues
- **Limitations**
 - Higher Cost
 - Temperature Limitations

SURFACE FINISH

- Long Period Waviness
- Short Period Waviness
- Local Defects (Pores, Etc.)
- Read Through (Welds, Ribs, Fibers)
- Quantitative Measurements

HEAT RESISTANCE

- E-Coat Temperatures (400F)
- Paint Oven Temperatures (280 - 325F)

SKIN MATERIALS

	<u>Mild Steel</u>	<u>B/H Steel</u>	<u>SMC</u>	<u>T/P</u>	<u>Al</u>
Cost	+	+	-	-	-
CTE	+	+	+	-	+
Surface	+	+	-	+	+
Stiffness	+	+	+	-	+
Formability	+	+	+	+	-
Joinability	+	+	-	-	-
Recyclability	+	+	-	+	+
Repairability	+	+	-	+	+
Lightweight	-	-	+	+	+
Dent Resistance	-	+	+	+	-
Heat Resistance	+	+	-	-	+

APPLICATION STRATEGIES

- Skin Panels

- Body Structures

- Engine Components

BODY STRUCTURES

MAJOR ISSUES

- Cost Effectiveness
- Lightweight
- Formability
- Functionality (Durability, Crash, NVH)
- Weldability/Joinability
- Repairability
- Recyclability

PRIMARY STRUCTURE

- Structural Integrity
- Sustain Primary Loads
- Manage Crash Loads
- Provide Acceptable Vehicle Dynamics (NVH)

BODY STRUCTURE MATERIALS

- Mild Steel
- High Strength Steels
- Aluminum Alloys
- Structural Composites (FRP)

BODY STRUCTURES POTENTIAL WEIGHT REDUCTIONS

- High Strength Steels (Up to 15%)
- Aluminum Alloys (Up to 40%)
- FRP Composites (30-50%)

BODY STRUCTURE

- High Strength Steels
 - Yield Strengths 35-75 ksi
- Advantages
 - Cost
 - Property Control (e.g. Continuous Anneal Lines)
- Limitations
 - Formability
 - Weight (?)
- Aluminum Alloys
- Advantages
 - Lightweight
- Limitations
 - Cost
 - Formability
 - Joinability (?)

BODY STRUCTURE

Aluminum Alloys -- Two (2) Types of Construction

- Stamped and Bonded Construction
- Extrusions for Space Frame Construction

STRUCTURAL COMPOSITE PROCESSES

- HSRTM
 - Maximum Part Integration
 - Optimum Weight/Performance
 - Process Requires Development
- Compression Molding
 - Experience
 - Existing Materials

BODY STRUCTURE

- Structural Composites (FRP)
- Advantages
 - Lightweight
 - Low Investment Cost
 - High Part Integration
- Limitations
 - High Volume Capability
 - Fully Accounted Cost (?)
- Derivative Vehicle Structures
- Complete Composite Body Shells

BODY STRUCTURE

- **Derivative Vehicle Structures**

- **Complete Composite Body Shells**

- **Derivative Vehicle Structures**

- **Complete Composite Body Shells**

BODY STRUCTURE

ULTIMATE OBJECTIVE

- Primary Structure With Class A Surface Quality

BODY STRUCTURE MATERIALS

	<u>Mild Steel</u>	<u>HSS</u>	<u>Al</u>	<u>FRP</u>
Cost Effectiveness	+	+	-	?
Lightweight	-	+	+	+
Formability	+	-	-	+
Joinability	+	-	-	+
Repairability	+	+	-	+
Recyclability	+	+	+	-

APPLICATION STRATEGIES

- Skin Panels
- Body Structures

- | |
|---|
| <ul style="list-style-type: none">• Engine Components |
|---|

ENGINE MATERIALS TECHNICAL TRENDS

- Materials Only One Aspect
- Alternate Materials Consideration At Design Inception
- Wide Variety of Newer Materials

LIGHTWEIGHT ENGINE MATERIALS POTENTIAL SYNERGIZED BENEFITS

- 5% Fuel Economy
- 25% Reduction in HC Emissions
- 50% NVH Improvement
- 10% Performance Improvement
- 35% Weight Reduction
- Improved Package Efficiency

GASOLINE ENGINE LIGHTWEIGHT COMPONENTS

Materials and Applications

- Titanium Alloys
(Valves, Retainers, Connecting Rods, Springs)
- Rapidly Solidified Aluminum Alloys
(Intake Valves, Connecting Rods, Retainers)
- Metal Matrix Composites
(Pistons, Connecting Rods, Retainers)
- Fiber Reinforced Plastics
(Piston Skirts, Connecting Rods, Retainers)
- Ceramics, Aluminides
(Valves)

ENGINE MATERIALS

Valve System

Lightweight valve system components allow lower spring loads which reduce friction -----> Fuel Economy Gains

<u>Component</u>	<u>Material</u>
Exhaust Valve	Titanium Alloy, Ceramics
Intake Valve	Ti, RS Al Alloy, Ceramics
Valve Spring Retainer	Titanium
Valve Springs	Titanium
Tappets	Al, MMC
Rocker Arms	Al, MMC, FRP

Power Conversion System

NVH and emissions improvements through weight reduction and lower crevice volume

<u>Component</u>	<u>Material</u>
Piston	MMC, Plastic Skirt
Piston Pin	MMC, Ceramic
Connecting Rod	MMC, RS Al Alloy, Titanium

ENGINE MATERIALS

Engine Structure

Weight, size, cost reductions and NVH improvement through use of innovative materials such as aluminum, magnesium and composites (metal or plastic matrix) for engine blocks, cylinder heads and manifolds.

ENGINE BLOCK MATERIALS

- Cast Iron -- Thin Wall
- Aluminum
- Magnesium
- FRP

FUTURE AUTOMOTIVE MATERIALS

Evolution and Revolution!

For the Material Scientist -- A Dream Come True!

AUSTEMPERING

Professor James P. Nagy

ERIE COMMUNITY COLLEGE
Main & Youngs Road
Buffalo, NY

PREREQUISITE	The student should have knowledge of the topic, Isothermal Transformation Diagrams.
OBJECTIVE	The object of this experiment is to observe the effects of nonequilibrium cooling of a carbon alloy steel. Various percentages of bainite and martensite will be produced in samples using the austempering heat treatment.
EQUIPMENT	Two heat treat furnaces Rockwell hardness tester Water quench tank 250 ML stainless steel beaker Tongs capable of handling small samples Tongs capable of handling a beaker half filled with molten lead Hack saw Pliers Metallurgical polishing equipment Metallograph or metallurgical microscope
SUPPLIES	3/8 inch (9.525 mm) diameter SAE 4140 steel bar stock Lead chips (approximately 1 kg) Cold mounting compound 2% nital etch Bailing wire
SAFETY EQUIPMENT	Safety glasses Heavy weight apron heat resistant gloves
SAFETY PRECAUTIONS	Students must wear their safety glasses, apron, and gloves when performing the heat treating part of this experiment. Students must wear safety glasses when etching the samples with the nital etch.

PROCEDURE

1. Set one heat treat furnace to 845 degrees Celsius
2. Set the other heat treat furnace to 400 degrees Celsius
3. Add a sufficient amount of lead to half fill the stainless steel beaker and place it in a furnace set at 400 degrees Celsius.
4. Each student will cut a sample from the SAE 4140 steel bar approximately 3 mm in thickness. Cut a groove on the edge of the sample about 1 mm in depth, and diagonally across the sample cut a second groove 1 mm in depth. Take a piece of bailing wire approximately 50 mm long and wrap it around the sample in the two grooves making a wire handle on the sample.
5. The students in the laboratory class will break up into four groups.

GROUP A - will austenitize their samples at 845 degrees Celsius for 15 minutes, then quench their samples in the lead at 400 degrees Celsius for 10 seconds, then water quench their samples.

GROUP B - will austenitize their samples at 845 degrees Celsius for 15 minutes, then quench their samples in the lead at 400 degrees Celsius for 30 seconds, then water quench their samples.

GROUP C - will austenitize their samples at 845 degrees Celsius for 15 minutes, then quench their samples in the lead at 400 degrees Celsius for 60 seconds, then water quench their samples.

GROUP D - will austenitize their samples at 845 degrees Celsius for 15 minutes, then quench their samples in the lead at 400 degrees Celsius for 400 seconds, then water quench their samples.

NOTE - When the sample is removed from the 845 degree furnace and quenched in the lead at 400 degrees, the sample must be handled by the bailing wire handle. If the sample is held by tongs, the tongs will shield the sample from the lead and give an inadequate quench.

6. Remove any oxide coating or decarb and hardness test the samples using the Rockwell C scale.

7. Mount the samples in cold mount, polish, and etch the samples.

REQUIRED

1. Observe the samples under the microscope and determine the percentage of bainite and martensite.

2. Make a table of hardness of samples and time in the lead quench.

3. Make a table of hardness and percent bainite in the samples.

4. Make graphs of hardness and percent bainite VS time in the lead quench.

5. Using the Isothermal Transformation Diagram show the cooling cycle for each sample.

LABORATORY INSTRUCTORS NOTES

The key to this experiment is to use a small sample of steel in order to get the sample to cool at the desired cooling rates. Wrapping a bailing wire around the sample to use as a handle to hold the sample is of great importance.

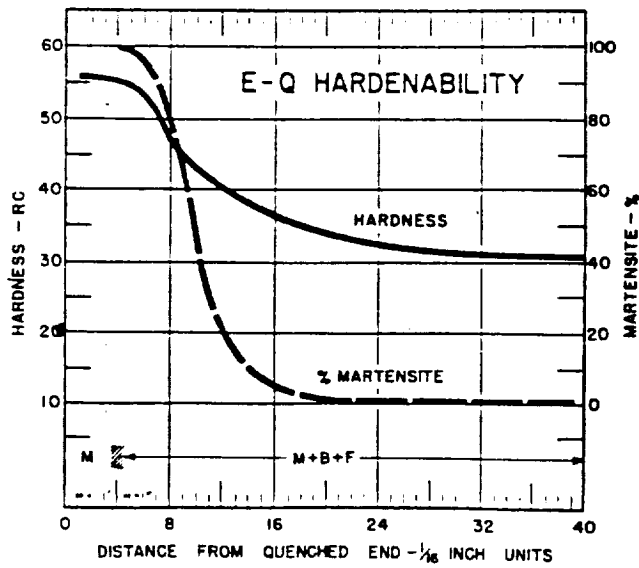
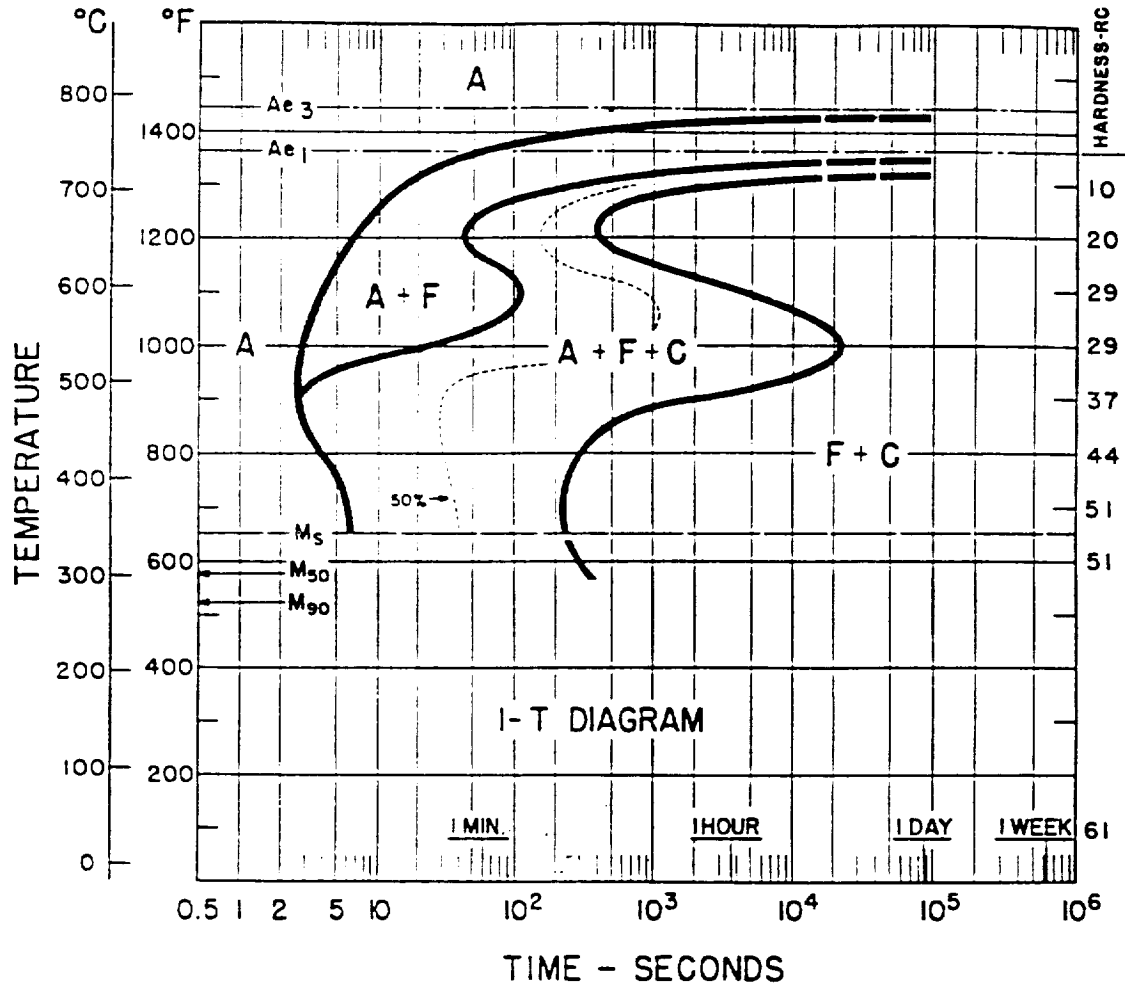
Have the students hardness test the samples before mounting them.

Steel will float in molten lead. Have the students make sure that the sample is not allowed to float.

The samples from group A will be all martensite. The samples from group B will contain just a slight amount of bainite. The bainite will etch much faster than the martensite and stand out, showing how the feathers of bainite are formed. The samples from group C will contain 30 to 50 percent bainite, and the samples from group D will be 100 percent bainite.

The bainite will show up best if the samples are lightly etched.

4140



4140

C-0.37 Mn-0.77
Cr-0.98 Mo-0.21

Austenitized at 1550°F

Grain Size: 7-8

LEGEND

A = Austenite M = Martensite
F = Ferrite B = Bainite
C = Carbide P = Pearlite

REFERENCES

Brick, Pense, and Gordon. Structure and Properties of Engineering Materials. Fourth edition, New York: McGraw Hill, 1977. p 136-152.

Pollack, Herman. Materials Science and Metallurgy. Fourth edition, Englewood Cliffs, NJ: Prentice Hall, 1988. p194-208.

Jacobs and Kilduff. Engineering Materials Technology. First edition, Englewood Cliffs, NJ: Prentice Hall, 1985. p193-199

Title:

Hands-On Thermal Conductivity

Presenter:

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Key Words:

Thermal Conductivity, Insulation, Heat, Energy, Convection

Prerequisite Knowledge:

Essentially none, this experiment can be used at any level higher than about fifth grade.

Objectives:

To convey to the students the correct interpretation of something they have probably all noticed in everyday life, and to demonstrate the difference in thermal conductivity shown by a variety of materials.

Equipment and Supplies:

Samples of materials exhibiting the widest possible range of thermal conductivities, all in blocks of approximately equal size, about 10 x 2 x 2 cm. A good basic assortment would be aluminum, glass, firebrick and Styrofoam, as representing high, medium, low and very low conductivities, respectively. A look at a table such as the one attached will reveal other choices which may be more available locally and which will represent other points along the thermal conductivity scale.

Procedure:

Arrange the test materials, with reference numbers, on a tabletop. The following morning, invite several (or all) students to briefly place the back of their hands in brief contact with the test blocks. They will notice that some of the blocks appear to be colder than others, and should note the comparative coldness of the blocks on simple data sheets. Remind the students that the materials have been on the tabletop overnight and could not possibly be at different temperatures. So why do some feel colder than others? The students should be allowed to speculate, and should record their speculations in their notebooks. The correct answer is that our hands are richly supplied with nerves which sense the passage of heat energy into or out of our bodies. Since the skin is normally at a temperature of about 28 C (80 F) and the blocks are perhaps 5 degrees C colder, the "warm" or "cold" feelings result from faster or slower energy transfer, respectively, from us to the test blocks. So metals such as aluminum feel cold, while good insulators like Styrofoam feel warmer. The students have probably all noticed that different substances in a room feel as if they are at different temperatures, but few have actually thought about it enough to know why this is true.

Sample Data Sheet:

This is obvious and will be omitted.

Instructor Notes:

Metals conduct heat better for the same reason that they conduct electricity better: their outer electrons are not localized but are shared by all of the atoms in the piece of metal. Their regular crystal lattice also helps the thermal energy, carried by vibrations called phonons, to travel better through metals. The glass does not possess this regular structure and is bonded such that outer electrons are localized and not shared. Much the same logic holds for the ceramic brick, except here conductivity is even lower because porosity has been intentionally left inside the brick. These pores are small and so do not support convection currents in the air filling the pores. This is the reason that fiberglass mat is good insulation; the fibers can fill space at a low density, meaning that not much material is present, and the fibers prevent formation of convective cells. Foamed plastics also make use of these tiny air bubbles to restrict the flow of heat. A piece of aluminum foil, crumpled very tightly, is a pretty good insulator, much better than solid aluminum, because of these small internal pores.

For energy efficiency, it is very important that houses be well-insulated. The material actually forming the insulation is not of much concern because its main function is to keep convection cells from forming and thus transferring heat from the inside wall to the outside wall. Since fibrous materials are very good at doing this, they should be non-flammable and non-toxic. Asbestos, a favorite natural insulation of the past, is being removed from many buildings at great cost because of its health hazard.

References:

Any college physics text will have a general explanation of thermal conductivity.

R. A. Flinn, P. K. Trojan, Engineering Materials and Their Applications, Third Edition, Houghton Mifflin Co., Boston, 1986, Pages 712-718.

J. A. Jacobs, T. F. Kilduff, Engineering Materials Technology, Prentiss-Hall, 1985.

Title:
Work-Hardening and Annealing in Metals

Author:
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Key Words:
Work Hardening, Annealing, Dislocations, Strain Hardening, Recovery, Stress Relief

Prerequisite Knowledge:
Students should have some introduction to dislocations and slip in metals.

Objective:
To demonstrate to the students, in a hands-on manner, how a metal (copper) becomes more resistant to deformation as it is deformed, and how annealing may be used to restore the ductility of the metal. The experience provides a means of making dislocations more real to the student, and the ensuing discussion shows positive and negative effects of the phenomenon.

Equipment and Supplies:
Each student is provided with two pieces of #10 bare copper wire, about 20 cm long, and the students share a pair of common pliers which are used to make the original bends in the wire. For the annealing, a furnace capable of 225 C is required. If none is available, a home oven capable of 450 F will do.

Procedure:
After bending each piece of wire according to the sketch on the next page, The students are asked to grasp the wire sections by hand, then to twist the center section of wire through three complete revolutions. The students will note that this is fairly easy at first, but gets quite difficult. Why is this? After some discussion, the students should be told that the copper was originally in a soft or annealed condition and is being work hardened by the twisting. Each student should twist both segments of wire because they are to be compared after one is annealed.

Place one wire from each student into a furnace or oven, and heat to 225 C (450 F) for at least 2 h; turn the oven off and allow the wire to cool inside. The next day, take the wire samples out and redistribute. Note the darkened color of the wires; this is caused by surface oxidation during annealing. Each student will now twist the non-annealed wire through one more complete turn, then do the same for the annealed wire. The difference in the effort required will be quite obvious. How did the heat affect the cold-worked metal?

Instructor Notes:
Beyond the use of normal care, there are no unusual precautions for this experiment. Before the experiment, the students need to be told about the role of slip in the deformation of metals, and to be briefly introduced to

dislocations as a way in which slip is made easier. Essentially, dislocations make it unnecessary to lift an entire plane of atoms and move it in reference to the plane below it. It may be helpful to use the analogy of the carpet; if a carpet is slightly misplaced in a room, it is not necessary to lift the whole thing at once to move it. A much easier way is to make a small bump in the carpet, starting a wall or corner, then simply push the bump across the room. The entire carpet can be moved easily this way. It might be effective to demonstrate this, using a small piece (1-2 square meters) of carpet remnant or sample.

The reason why the copper becomes work-hardened is that the dislocations, which are originally fairly few in number, increase in number as deformation continues and get tangled with each other and with grain boundaries in the metal so that moving them becomes increasingly difficult. Annealing provides energy which can be used to move the dislocations out of the metal so that it can once again be deformed easily.

Positive Aspect: For metals that are to be used only at low temperatures where annealing cannot occur, cold-working can be used to increase the resistance to deformation. Metals differ in their sensitivity to work-hardening; copper was chosen because it work-hardens well and because it can be annealed at a relatively low temperature.

Negative Aspect: A work-hardened metal is more subject to breaking during forming than an annealed one. For example, metals are made into wire by pulling them through a series of holes in a die, each hole smaller than its predecessor. After most metals have been pulled through these holes, they work-harden to the point where they must be annealed before further forming is done or they are likely to break.

References:

R. A. Flinn, P. K. Trojan, Engineering Materials and Their Applications, Houghton Mifflin, 1986

R. E. Reed-Hill, Physical Metallurgy Principles, Van Nostrand, 1964

Sources of Supplies:

Bare copper wire is commonly used as a ground wire in electrical circuits, and can be obtained at any electrical supply house for about \$0.40/meter. #12 Wire is even cheaper and could be used if #10 is not available, but it will twist much easier and the difference in effort when work-hardened may be harder to detect.

CERAMIC FIBERS

Bruce M. Link

Purpose

The purpose of this experiment is to demonstrate that glass ceramics are fundamentally stronger than everyday observation leads us to believe.

Acknowledgments and Intentions

This paper does not put forth any new ideas or particularly unique or original work, but rather is an assimilation of various text materials. It draws heavily upon basic work conducted by Griffith, Jurkov, Anderegg and others who experimented with ceramics in the early 1900's. The objective of this paper is to present an experiment that will readily demonstrate some of the basic properties of glass and ceramics.

Materials

- 2 Small "C" clamps balanced and drilled as in figure 1.
- 1 Spool of fine high purity aluminum wire (22 gage 17 stranded should work well. The finer the better)
- 1 Plastic bucket or other suitable lightweight container.
- 1 Precision scale good to 5g over a 10Kg range.
- 10 Kg of sand or other heavy substance. (lead shot will do)
- 10 Glass rods
- 1 Bunsen burner, gas supply and striker.
- 1 Micrometer good to .0005"
- 1 Safety glasses

Experimental Setup

Basically the two "C" clamps will be used to apply tension to either glass or metal fibers. One "C" clamp will have to be suspended from the ceiling or other suitable support structure. The second "C" clamp will be affixed to the bottom of the fiber and the bucket (or pannier) suspended from it. Weight will be added to the pannier until the fiber breaks. The diameter of the fiber will then be measured by means of the micrometer. Figure 2 shows the experimental setup.

Procedure

Aluminum Wire

1. Trim off a piece of aluminum wire approximately 50mm in length. Strip off the insulation and separate a single strand. Make several measurements along its length and determine its average diameter.
2. Place the strand in the setup as shown in figure 2. Add weight until the fiber breaks. Measure the diameter of the shorter of the two pieces at the break. Determine the load on the fiber. Calculate and plot the breaking strength (weight/unit area).
3. Set the longer fiber up in the setup and repeat steps 2 and 3 until the longest fiber is too short to continue.

Glass Fiber:

1. Take one of the glass rods and heat it slowly and carefully over the Bunsen burner. Slowly draw out a glass fiber that is approximately 0.010" in diameter and 50mm long. Trim the fiber from the rod and place it in the fixture. Repeat steps 2 and 3 from the aluminum wire experiment.

Part II

1. Produce four fibers each of aluminum and glass as in Part I. Trim the fibers to the following lengths: 5mm, 10mm, 20mm and 40mm.
2. Measure the initial breaking strength of each fiber and plot the results.

Results

The results of part I of the experiment will show that the apparent breaking strength for both the glass fiber and the aluminum fiber increases as the procedure is repeated. The results for glass should be much more dramatic.

The results of part II of the experiment show something very different about the glass and the metal fibers. In both cases the strength of the fiber will tend to decrease as the length increases, but the curve for the aluminum fiber is much flatter than that for the glass fiber.

Discussion

It is possible to overlook the differences between the results for the glass and metal fibers and to say that the general trends are the same. If you do, you will not discover anything about glass. The questions that this experiment raises are

1. In part I why does the strength of the glass improve so radically? Why doesn't the metal strength improve as dramatically?
2. From part II why is it that the length of the fiber should have such a crippling effect on the strength of the fiber?

It is easy to explain the increase in the strength of the aluminum fiber from part I of the experiment as being due to work hardening of the entire fiber due to repeated loading. The measurements of the fiber diameter should be enough to convince you that this is taking place. However, the improvement in glass fiber strength cannot be explained by the same theory. If one looks at the measurements of the glass fiber diameter at the places where it broke, one should note that the fiber tends to maintain its average diameter. The metal fibers continually neck down.

Part II of the experiment provides a small clue to the breaking of glass fibers. If one's data is very good (this can be accomplished by repeating the experiment many times and averaging the results), one should be able to calculate the breaking strength of a glass fiber of zero length. It should be noted that this value will be much higher than that for the aluminum fiber. If a material is so theoretically strong, how is it that it can be so weak? An explanation for this can be helped along by taking one of the glass rods and slamming it against the table. Glasses are indeed brittle and non ductile. If one then imagines that there is a very small crack in the glass (a Griffith crack or one even larger), then this crack will

concentrate the stresses in the glass. Since the glass cannot ductilely deform to spread the stress very well, the stress will remain high and cause the crack to propagate. Metals, on the other hand, can ductilely deform and reduce the localized stress. This makes them more resistant to internal defects.

This line of reasoning also neatly explains the glass sensitivity to length. The shorter the fiber is, the lower is the probability of its having a serious defect (and almost any defect is serious for a ceramic in tension). The aluminum fiber will be less sensitive to length because it can plastically deform and negate the seriousness of most defects.

References:

1. Physical properties of glass, J.E. Sanworth, Oxford at the Clarendon Press, 1950.
2. "Ind. Eng. Chem.," 1939,31,290, F.O. Anderegge.
3. "Phil. Trans. Roy. Soc.," Series A 1920,221,163, A.A. Griffith.

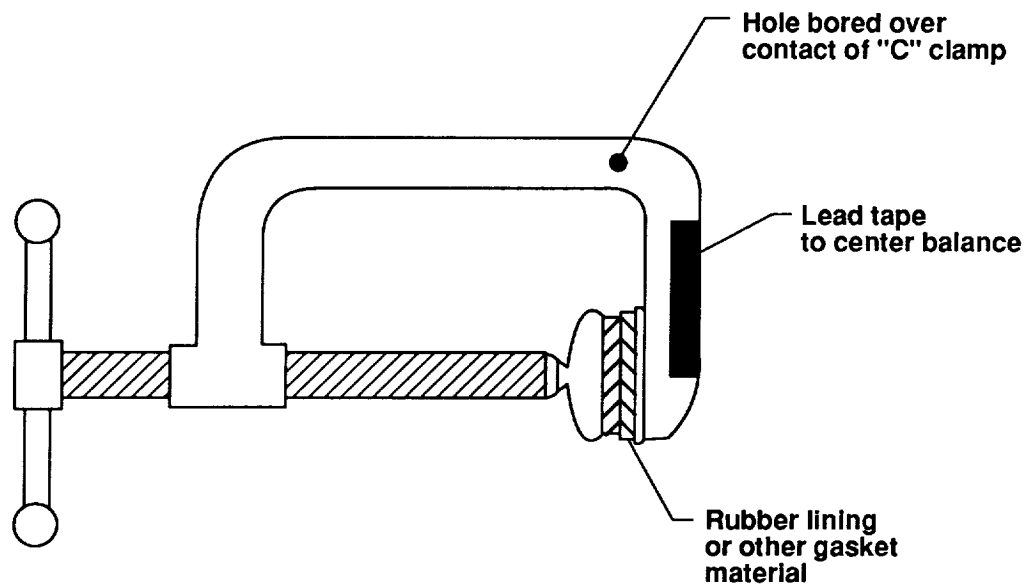


Figure 1 "C" Clamp Construction

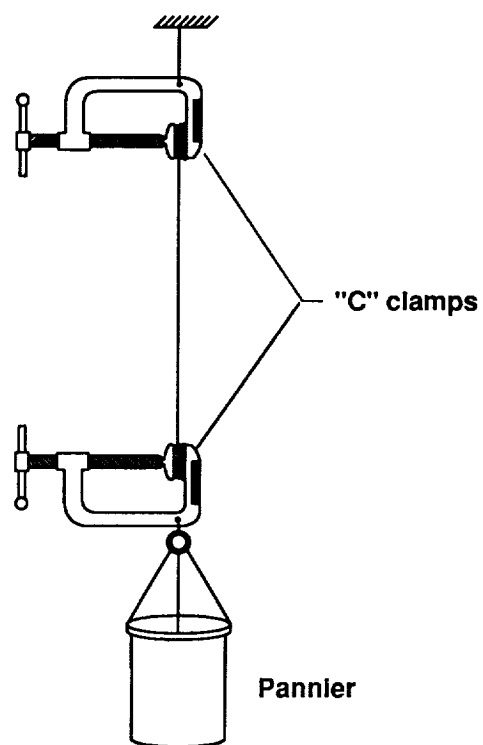


Figure 2 Experimental Setup

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